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The rapid template fabrication process for producing a LIGA heat exchanger bonded to a mechanical seal

Jason Patrick Tuma
Louisiana State University and Agricultural and Mechanical College, jtuma1@lsu.edu

THE RAPID TEMPLATE FABRICATION PROCESS FOR PRODUCING A LIGA HEAT EXCHANGER BONDED TO A MECHANICAL SEAL

A Thesis

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

ABSTRACT

Mechanical seals are widely used in the chemical processing, aerospace, and automotive, industries. When a mechanical seal fails, considerable cost is typically involved in replacing the seal. Most seal failures are a result of high temperatures generated by frictional heating. Effectively removing the heat generated would significantly increase the mechanical seal's work life and lead to significant monetary savings.

One method of removing the heat generated is bonding an effective LIGA micro heat exchanger to the mechanical seal and forcing coolant through the heat exchanger. Previous work on this subject revealed promising results, especially for micro heat exchangers with microstructures taller than 500 μm. This previous work relied on PMMA templates that were lithographically patterned at CAMD by a synchrotron radiation source. These templates were 500 μm thick and patterned with an electron energy of 1.4 GeV. In this effort, 1000 μm tall templates were desired and had to be produced at CAMD's current electron energy of 1.3 GeV. The combination of thicker templates and lower electron energy made numerous lithographically patterned PMMA templates impractical. Therefore, a new template fabrication process was developed that only required one lithographically pattern template. Another issue of the previous work was that the adhesion strength of the micro heat exchanger was relatively weak and needed to be improved.

The purpose of this thesis is to present how a fabrication process consisting of silicone casting, machine wax casting, and electroforming can result in producing a LIGA heat exchanger bonded to a mechanical seal with the single use of CAMD's synchrotron radiation source. This thesis will also state how the adhesion between the micro heat

exchanger and seal was strengthened through surface treatments and electroplating conditions. The surface treatments included the use of sandblasting, activation, and Wood's strike procedure. The electroplating conditions included setting the electroforming surface at an inclined orientation and applying a relatively low current density of 5 mA/cm^2 to the electroforming surface.

CHAPTER 1. INTRODUCTION

1.1. Research Motivation

Mechanical seals are widely used in the chemical processing, aerospace, and automotive, industries. When a mechanical seal fails, considerable cost is typically involved in replacing the seal. In extreme cases, environmental hazards may occur and production equipment worth tens of millions may be shut down in order to replace the seal. Frictional heating, generated at the dynamic seal interface, raises the temperature of the seal significantly. This temperature rise causes, for a variety of reasons, most seal failures.

To prevent temperature related seal failures, coolant must be supplied in close proximity to the seal interface and the net thermal resistance coupling the coolant and heat source must be minimal. This ensures that the rise of the seal face temperature will be minimal during operation. This design philosophy was incorporated in a mechanical seal prototype [1] shown in Figure 1-1 that was fabricated at LSU and used a pin fin micro heat exchanger located beneath the load bearing surface to very effectively cool the seal during tribometer tests.

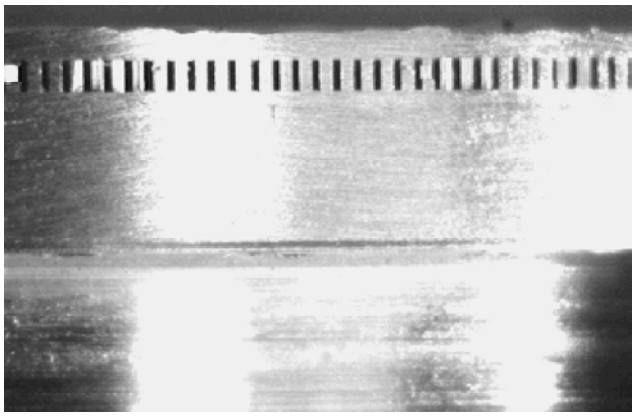

Figure 1-1: Seal Prototype

The success of the first prototype led to a redesign of a next generation mechanical seal with active cooling using a micro heat exchanger. This new prototype design specified a field of micro pins 1000 μm in height. The height of the pin fins in the original prototype was only 500 μm in height. This seemingly small change in design had important consequences with regards to fabrication. To fabricate the original prototype, the LIGA (a German acronym for Lithographie, Galvanoformung, Abforming) process was used at CAMD (Center for Advanced Microstructures and Devices) to perforate holes in a sheet of Polymethyl methacrylate (PMMA) with thickness 500 μm. The synchrotron radiation source used to perforate the template was operating at an electron energy of 1.4 GeV. This PMMA sheet then served as a template in a subsequent electroforming process to produce the desired heat exchanger on the end face of a mechanical seal. However, when the height of the pin fins was increased to 1000 μm, it was no longer feasible to produce PMMA templates. The combination of increased height and CAMD's current electron energy of 1.3 GeV made the exposure times prohibitive. Therefore, a practical method needed to be found to create templates to electroform the taller features. The goal of this research is to fabricate a micro heat exchanger using non-conductive templates that could be rapidly supplied without the repeated need of a synchrotron radiation source.

1.2. Mechanical Seals

Mechanical seals are often used to seal fluids in pumps and turbines where the rotating shaft enters an enclosure. Pump housings and transmission boxes are typical applications. The Figure 1-2 shows a schematic representation of a rotating shaft in a pump housing.

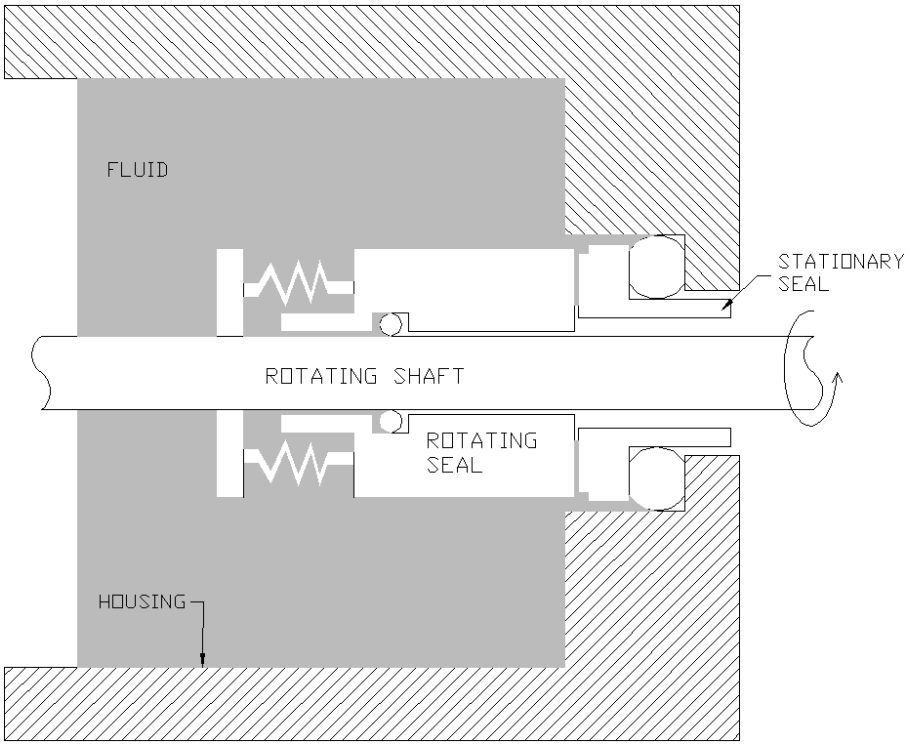

Figure 1-2: Schematic Representation of a Mechanical Seal Face

The rotating component of the dynamic seal is attached to the shaft, whereas the stationary component is mounted on the housing. The O-rings (also called the secondary seals) prevent the fluid from leaking between the shaft and rotating component of the seal as well as between housing and stationary component of the seal. Axial loading from the spring and sealed fluid pushes the two components of the seal together. The dynamic seal occurs at the interface of both rotating and stationary component of the seal, where the rotating face slides relative to the stationary face.

When the rotating face slides relative to the stationary face, the rate of heat generated is equal to the frictional force multiplied by the velocity of the rotating face. Quite often this frictional heating results in non-uniform seal temperatures that result in a variety of fatigue problems, ultimately leading to pitting, cracking, and the ultimate

failure of the seal. Lowering the operating temperature prevents these physical phenomena and results in lower operating costs, continued production profits, and fewer environmental emissions.

1.3. Previous Micro Heat Exchanger/HARMs Production

The seal prototype shown in Figure 1-1 was fabricated from a procedure that was based on the LIGA produced template [1]. The steps to produce the LIGA template are as follows and shown in Figure 1-3. The LIGA process uses a photoresist such as SU-8 that is positioned behind a mask. A collimated X-ray beam is applied to a mask that selectively allows x-rays to pass to the photoresist. (Step one of Figure 1-3) The photoresist's molecular structure changes as a result of the exposure to the x-rays. The photoresist is then immersed into a developer that eradicates the areas with the modified molecular structure. (Step 2 of Figure 1-3) The resulting photoresist template is then used to electroplate HARMs onto the substrate. (Step 3 of Figure 1-3) The electroplated microstructures could be the final product (step 4 of Figure 1-3) or serve as a mold. This mold could then be inserted into an injection molding or embossing machine to repeatedly reproduce a secondary polymer template with the exact geometry of the primary photoresist template (Step 5 of Figure 1-3)

Steps 1 and 2 were used to produce the seal prototype template. The required template consisted of a template of 500 μm thickness with a square array of perforated square holes with side width, a=175 μm and edge-to-edge spacing of w=110 μm. PMMA was exposed and developed to form the template used for the final product. The resulting template was directly clamped onto the end face of an annular ring of the jig, as shown in Figure 1-4.

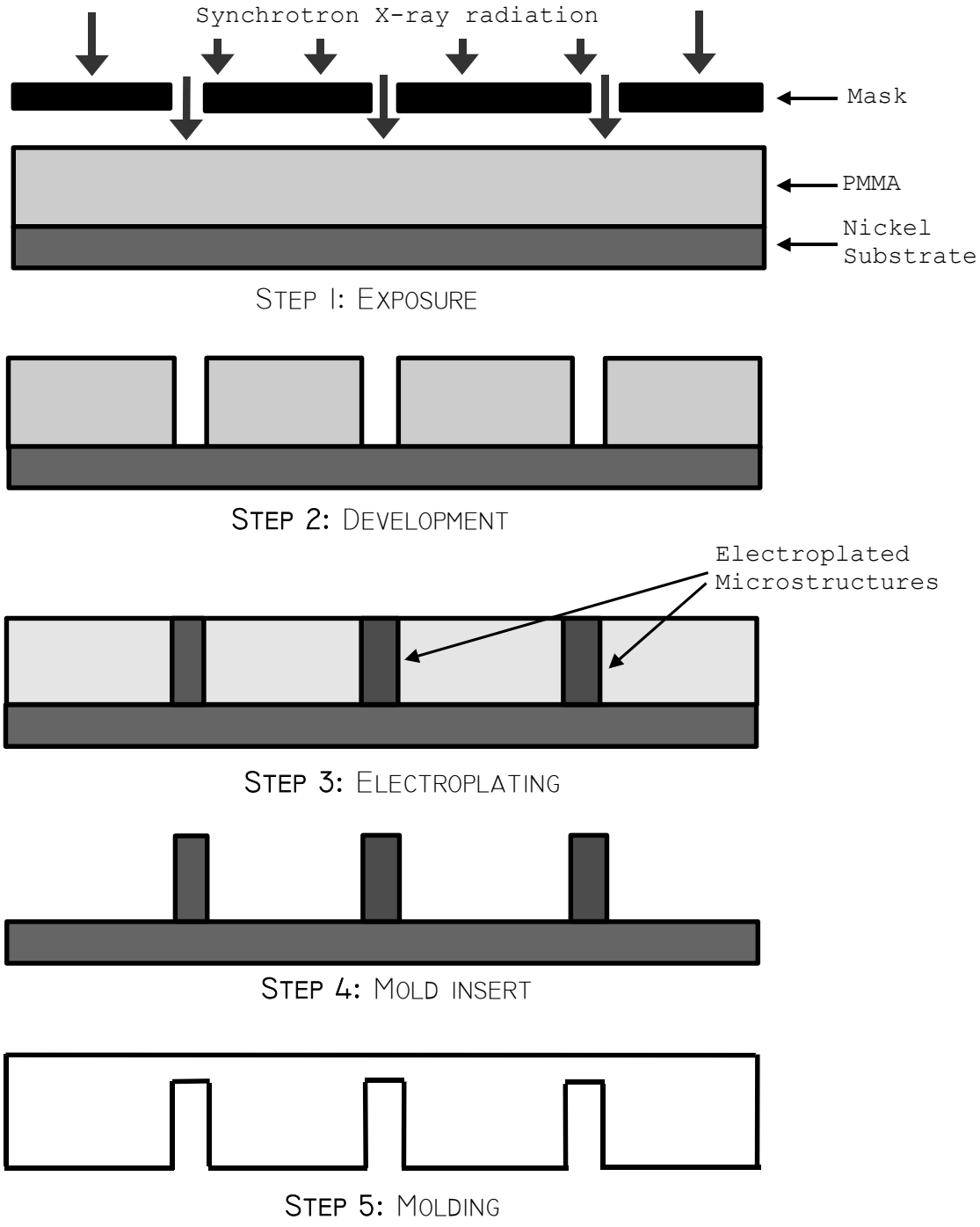

Figure 1-3: LIGA Steps [2]

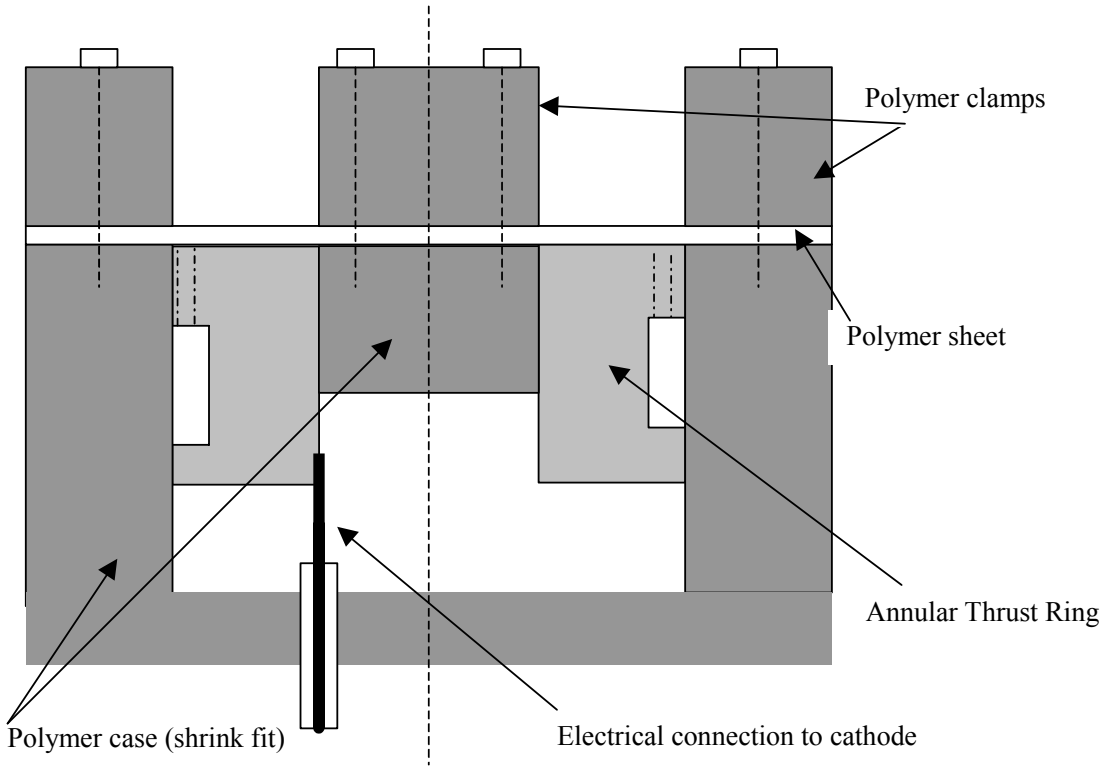

Figure 1-4: Jig to Hold Substrate for Electroplating

The jig used shrink fits to encase the inner and outer radii a stainless steel ring in a polymer casing so that the end face is exposed. The jig was immersed into an electroplating solution. Nickel was electrodeposited onto the end face of the thrust ring through the perforated holes and into the perforated holes of the template. After the perforated holes in the template were filled with electroplated material, the electroplating process was no longer geometrically bound by the template. However, electroplating continued and resulted in the electrodeposited structures merging. This eventually formed a new surface (overplating) that is parallel to the end face from which the plating originated (substrate). The overplated surface becomes a load bearing cover of the micro heat exchanger for this application. Cooling fluid can be forced through between the HARMs to cool the load-bearing surface. [1]

1.4. Template Production Alternatives

To produce 1000 μm microstructures, the template fabrication process needed to be modified because repeated exposures of 1000 μm PMMA was not feasible. Instead, repeatable template production was needed that did not involve the LSU/CAMD synchrotron radiation source. This dictated the use of some type of injection molding/embossing/stamping/casting process using a mold. Injection molding set-up is relatively extensive compared to embossing/stamping/casting methods and was never examined as an alternative to any detail. The Microsystems Engineering Team (μSET) at LSU had extensive expertise embossing PMMA sheets, and this seemed a promising method to manufacture the needed templates. Unfortunately, post-machining difficulties using embossed PMMA templates generated the need to find another alternative method to produce the needed templates. Wax casting using LIGA-fabricated *silicone* molds was investigated as a method to produce the needed templates. This method of template fabrication proved successful.

Other investigators have used silicone molds to fabricate templates for a variety of MEMS applications. A two-part silicone called poly-dimethylsiloxane (PDMS) has shown the potential for rapid replication of HARMs. [3],[4] PDMS can be mixed and cast onto a LIGA produced mold insert. The PDMS and mold insert are placed in a vacuum during or immediately after casting to remove any trapped air pockets. After vacuuming the PDMS, it is allowed to cure and then peeled off the mold insert. This results in a silicone template similar to the PMMA templates produced through embossing and mold injection techniques.

The silicone mold is then deposited with sacrificial layers of metal (typically gold or titanium/gold 2 μm thick) on the surface by using sputtering or thermal evaporation techniques. Nickel is electrodeposited onto the sacrificial layer. The PDMS is then removed once the electrodeposition is completed. The sacrificial layer is then carefully etched away from the replicated mold insert. The over-electroplated region is then planarized as desired. [3]

This procedure leads to the rapid replication of HARMs without the use of a synchrotron radiation source. However, the HARMs are free standing, i.e. not bonded to a substrate. This procedure is not easily incorporated into manufacturing of the micro heat exchanger bonded to the mechanical seal. But, the idea of utilizing a silicone mold with micro posts for required template was attractive for several reasons. Using a silicone mold prevents the LIGA mold insert from experiencing degradation that typically occurs with injection molding/embossing/stamping/casting processes. Multiple silicone molds can also be produced from the one LIGA mold insert. With multiple silicone molds, the template production could be increased to meet any practical demand.

Template material issues and preparation for electroplating still needed to be solved. Suggestions from [5] indicated that machine wax commonly used by machinist could be a candidate for the template material because of its trouble-free mold formation, machinability, chemical properties, and removal.

1.5. Research Goals

The production of a template that allowed the rapid repeated fabrication of a mechanical seal with a LIGA micro heat exchanger bonded to it was required. To accomplish this, the following goals needed to be achieved:

1.) Produce a silicone mold with micro posts from a LIGA mold insert

2.) Fabricate a machinable wax templates from the silicone mold

3.) Electroform the micro heat exchanger using the machinable wax template

4.) Achieve a high bond strength between the seal and micro heat exchanger

To satisfy goal number one, a mold insert previously fabricated at LSU was used. This mold insert contained a circular mesh with micro holes. A two-part silicone was selected and cast onto the mold insert. A vacuum was applied to remove any trapped air pockets and the silicone mold was allowed to cure.

To satisfy goal number two, molten machine wax was poured onto the silicone mold without a vacuum. The molten machine wax would vaporize under vacuum and create greater void spaces. In order to remove trapped air pockets, the silicone posts were gently rubbed with a blunted knife until the wax began to solidify. After reaching room temperature the silicone mold was peeled away. The machine wax template was then machined down to the correct template height such that the micro holes would become the necessary through holes.

Goal three was achieved by clamping the wax template to a similar substrate jig as shown in Figure 1-4 by [1]. The template and substrate jig were then placed into a nickel sulfamate bath where the electroforming of the micro heat exchanger could take place.

Goal four was achieved by physically and chemically treating the plating surface of the seal before electroforming occurred. Two physical treatments were involved to

improve the bond. The first treatment was sandblasting the plating surface to increase the surface area and enhance mechanical locking of the electroplated nickel. The second physical treatment was allowing underplating (electroplating beneath the wax template) to increase the bond surface area between the electroplated nickel and substrate. The chemical treatment required an activation process and application of a thin layer of nickel (Wood's strike) before electroplating. The activation and Wood's strike promoted the optimal conditions for the nickel to be electrodeposited on the atomic level. In addition, a modified jig was built to promote nickel deposition bonding and uniformity during electroplating.

CHAPTER 2. TEMPLATE FABRICATION PROCESS

2.1. Silicone Mold Fabrication

The first step of producing useable wax template is manufacturing a silicone mold with the essential material properties and geometry for wax castings. The silicone material has to withstand 130˚C temperatures and maintain its durability after repeated uses. The silicone material selected for this application was the RP 134 SI, a silicone manufactured by Vantico, Inc. The RP 134 SI was selected because of its high tear strength [6] and the fact that it had been used successfully to produce wax castings by other μSET engineering team members.

The silicone mold's geometry is established by using a mold insert and a PMMA jig. A mold insert previously constructed by Ryan Turner at LSU [7] was selected and is shown in Figure 2-1. The mold insert's mesh of microholes is used to form the geometry of the microposts of the silicone mold as shown in . The microhole mesh consisted of an inner diameter of 20.2 mm and an outer diameter of 34 mm.

Figure 2-1: Mold Insert Used to Produce Silicone Mold

Figure 2-2. shows a magnified view of the micoholes.

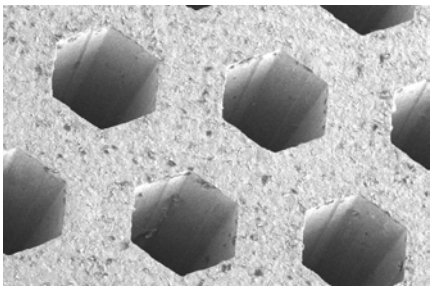

Figure 2-2: Magnified View of Mold Insert Microholes

The PMMA jig is used to construct the silicone mold's remaining geometry as also shown in and Figure 2-5.

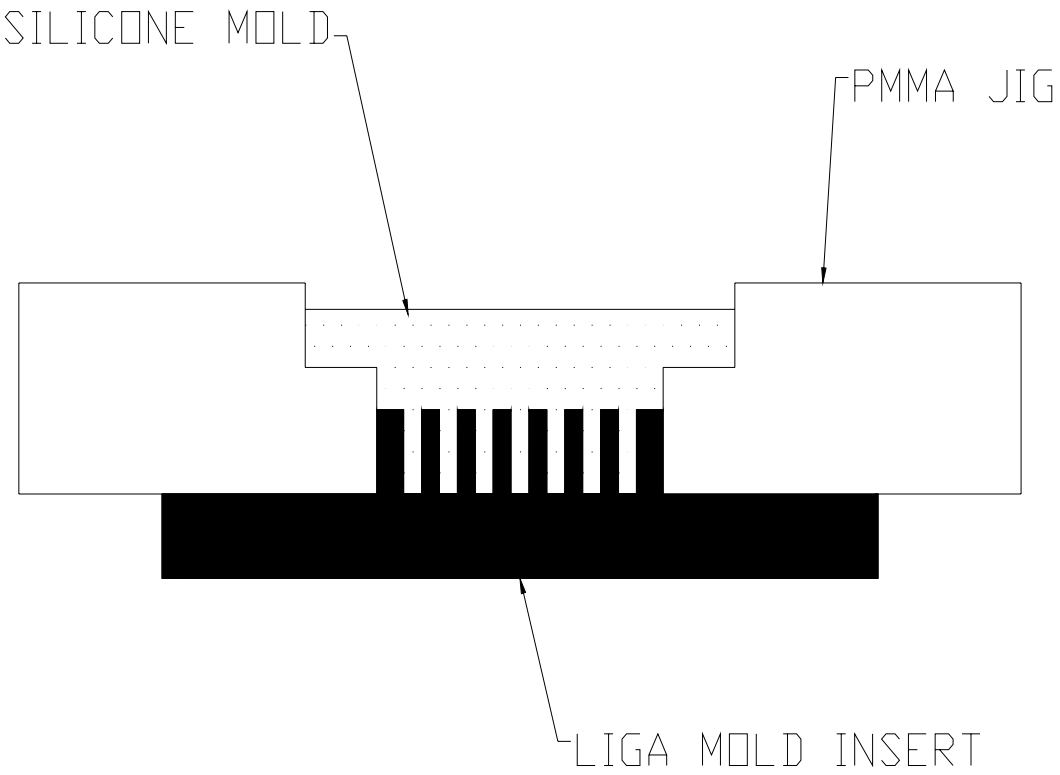

Figure 2-3: Cross-Section of Silicone Mold Production

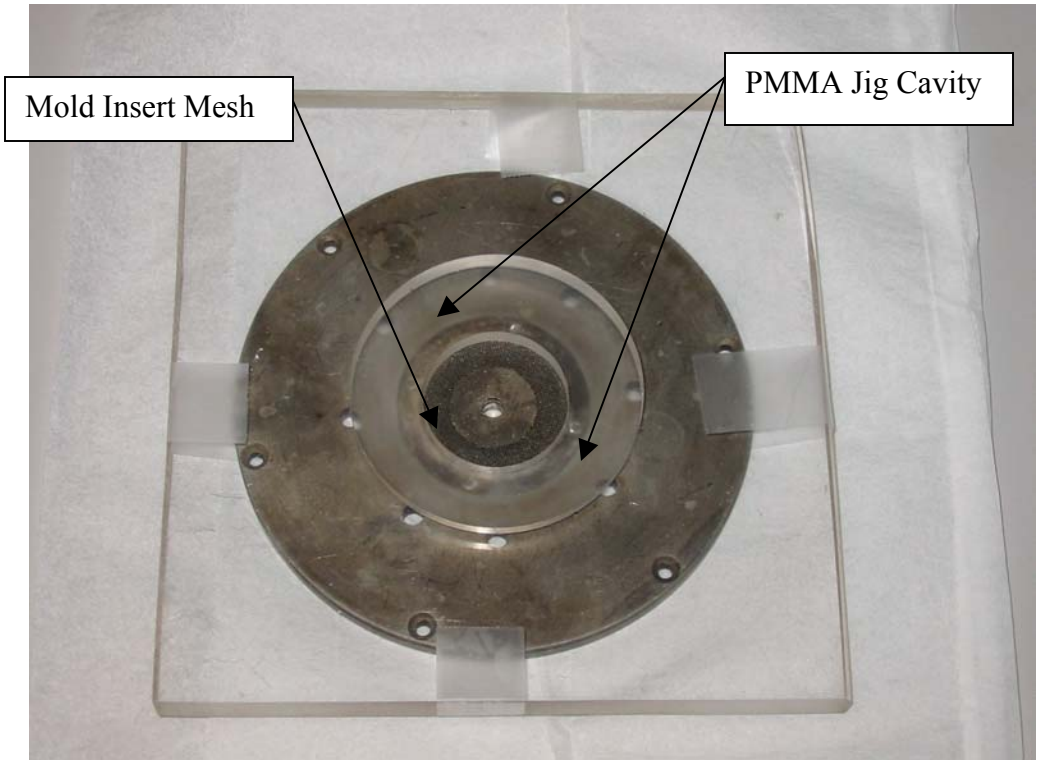

Figure 2-5: Mold Insert Clamped to PMMA Jig for Silicone Mold Production

With the mold insert clamped to the PMMA jig, the silicone mold production could proceed. The RP 134 SI is prepared according to the manufacturer specifications for pouring into the mold insert mesh and PMMA jig cavity. The mold insert mesh and PMMA jig cavity are coated with Eject Mold Release #32, a demolding agent, before the silicone was poured. The Eject Mold Release facilitates the silicone mold's removal from the mold insert. The silicone mixture is then poured onto the mold insert mesh and PMMA jig. The mixture, mold insert, and PMMA jig are immediately placed into a vacuum chamber to remove trapped air pockets as shown in Figure 2-6. A vacuum of 28.5 in Hg was achieved and cycled every ten minutes for forty minutes.

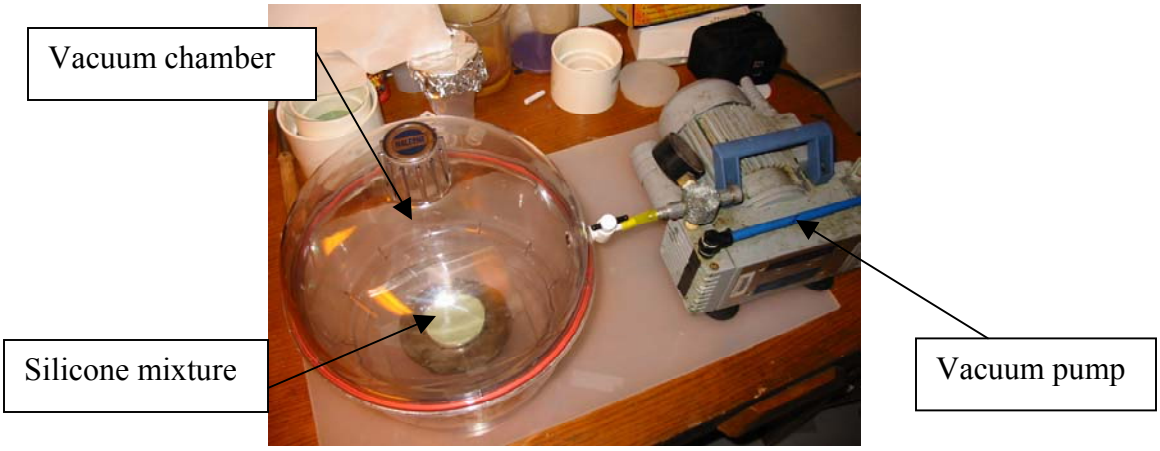

Figure 2-6: Silicone Mixture under Vacuum

After the vacuuming is completed, the mold insert, PMMA jig, and silicone mix are placed into an oven at 60ºC for at least twelve hours to ensure complete curing of the silicone. The mold insert, PMMA jig, and silicone are then separated from each other. The result was the silicone mold shown in Figure 2-7 and Figure 2-8.

The use of the PMMA jig forms a silicone mold with a flat surface along the silicone mold base as also shown in Figure 2-7. The flatness of the silicone mold base is a critical property for machining purposes and will be discussed in greater detail in section 2.3.

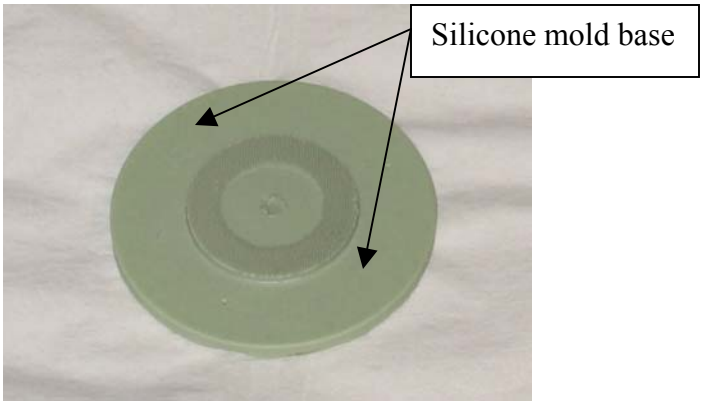

Figure 2-7: Manufactured Silicone Mold

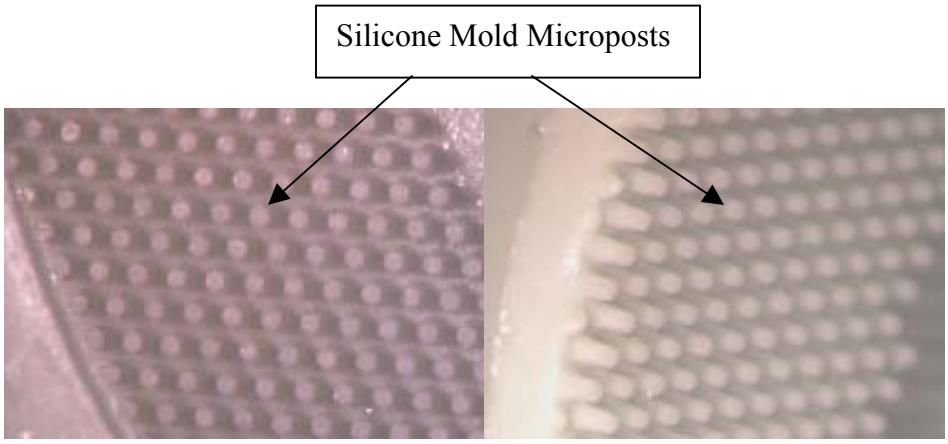

Figure 2-8: Top View (Left) and Angled View (Right) of Silicone Mold

2.2. Wax Molds

The wax material chosen to produce the wax molds was Freeman Machinable Wax available for purchase through MSC® Industrial Supply, Inc. This material was chosen due to three critical properties. First, it is easy to machine. Secondly, the machine wax maintains a satisfactory strength and rigidity at 55ºC. The strength and rigidity at this temperature is important because the wax template will be placed into an electroplating bath at 55ºC. Any softening of the wax template at this temperature would lead to distorted microfeatures and could not be tolerated. Thirdly, the machine wax does not dissolve when exposed to acetone like most wax materials.

In order to produce the wax molds, masking tape is applied the outer edges of the silicone mold as shown in Figure 2-9, providing a container into which the wax could be poured. The height of the wax mold is dictated by the amount of wax that is poured into the taped silicone mold. The height of the wax mold at this point is not a critical

dimension because the height will be reduced through later machining. Therefore, this dimension is allowed to have some variation.

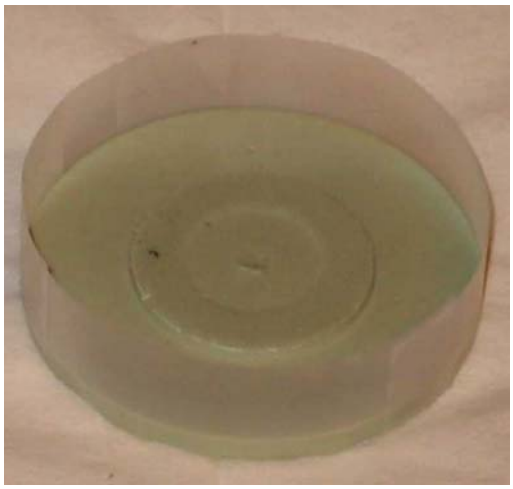

Figure 2-9: Taped Silicone Mold Ready to Accept Molten Wax

To achieve high quality wax castings, the following simple procedure is used:

1. The machine wax was melted in an oven at 130˚C.
2. The silicone mold and blunted knife were placed into the oven at 130˚C for five minutes. Both were removed from the oven, and the masking tape was immediately applied to the edge of the silicone mold.
3. To assist in the separation of the wax mold from the silicone mold, the Eject Mold Release #32 was applied to the silicone mold (for approximately two seconds) before pouring the wax
4. The melted wax was easily poured onto the silicone mold as shown in Figure 2-10.
5. The silicone mold's microposts were then gently rubbed with a blunted knife as shown in Figure 2-11. The rubbing action releases trapped pockets of air.

Vacuuming was not used to remove the trapped air bubbles because the melted wax vaporizes when placed under vacuum.

This procedure did not require any additional jigs to be machined. Furthermore, it was found that jigs that applied any stress to the silicone mold would distort the mold and produce non-flat castings that were of no value. The simple use of the masking tape allowed the silicone mold's base to retain its critical flatness for later machining. The use of the masking tape offers the additional final advantage of eliminating issues related to separation of the silicone and wax molds from the jig. Figure 2-12 shows the wax mold after separation from the silicone mold.

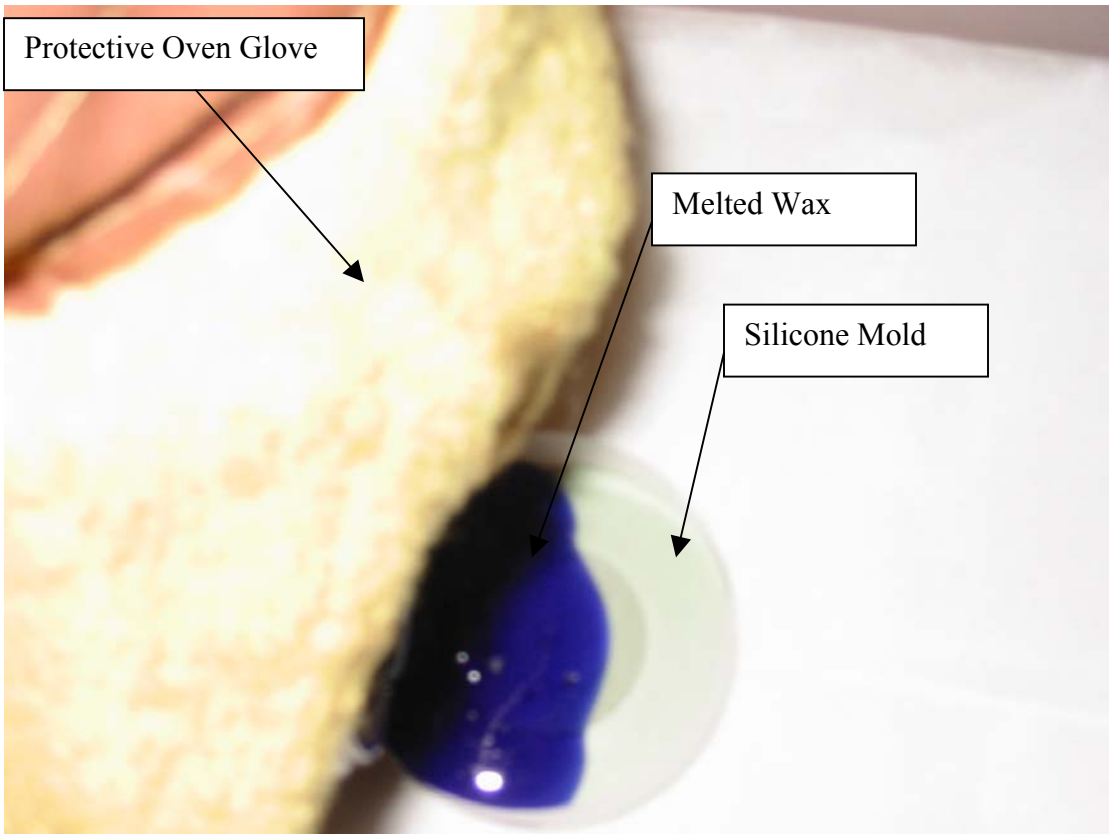

Figure 2-10: Wax Pouring onto Silicone Mold

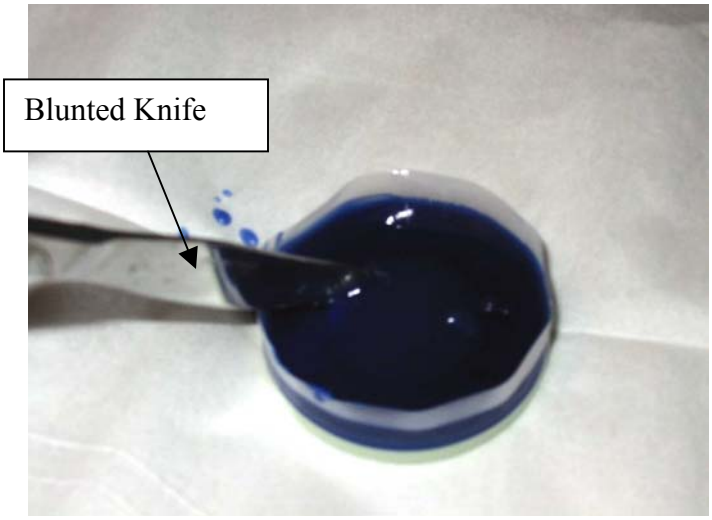

Figure 2-11: Rubbing Action to Release Trapped Air Pockets

Figure 2-12: Wax Mold/Template before Machining

2.3. Wax Mold Machining Issues

Once a wax mold is cast, the wax mold contains a recess with microholes as shown in Figure 2-13. In order to use the wax mold as a template for electroplating, the wax height must be reduced so that the microholes become through-holes as shown in Figure 2-14.

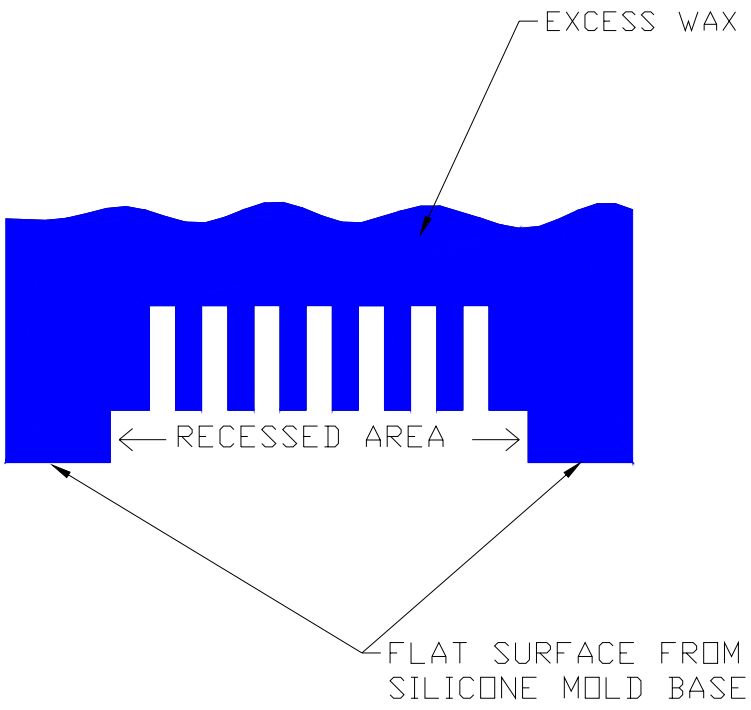

Figure 2-13: Wax Mold Cross Section after Casting

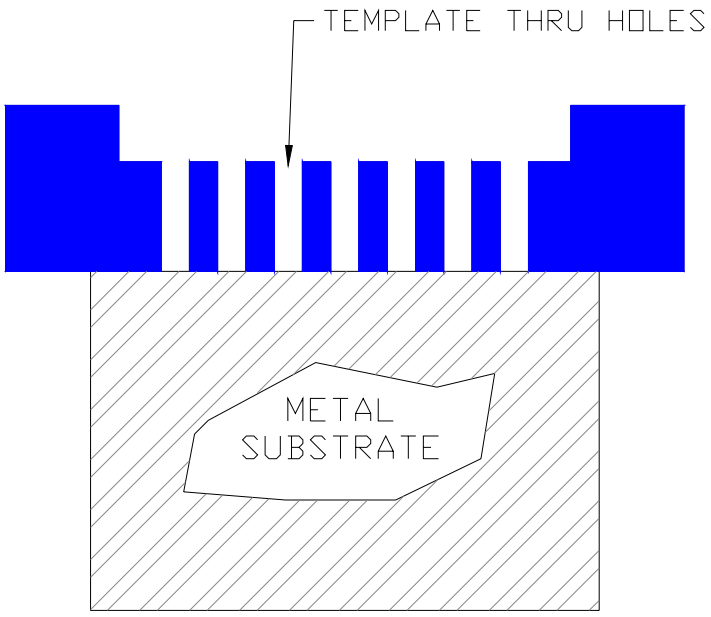

Figure 2-14: Desired Cross Section of Template Geometry on Metal Substrate

In order to remove the excess wax, the surface with excess wax was machined on a milling machine by flycutting. In this process, the wax mold's flat surface is held flat against a PMMA jig, shown in Figure 2-15 and Figure 2-16. 3M® double-sided tape of ½" width was used to hold the wax mold stationary throughout the fly-cutting process. Without the wax mold's flat surface, the double-sided tape would not have enough surface area and strength to hold the wax mold stationary.

Minor physical blemishes would often occur on the wax mold's flat surface that would prevent a flush interface between it and the double-sided tape. Two physical modifications are made to the wax mold's flat surface to eliminate theses blemishes. First, the flat area of the wax mold is sanded with 400 grit sandpaper. The sanding smoothes any blemishes that would inhibit adhesion to the double-sided tape. Second, a blade is use to remove a thin raised edge along the wax mold's outer diameter. This raised edge was due to gaps between the silicone mold and masking tape when the wax was poured.

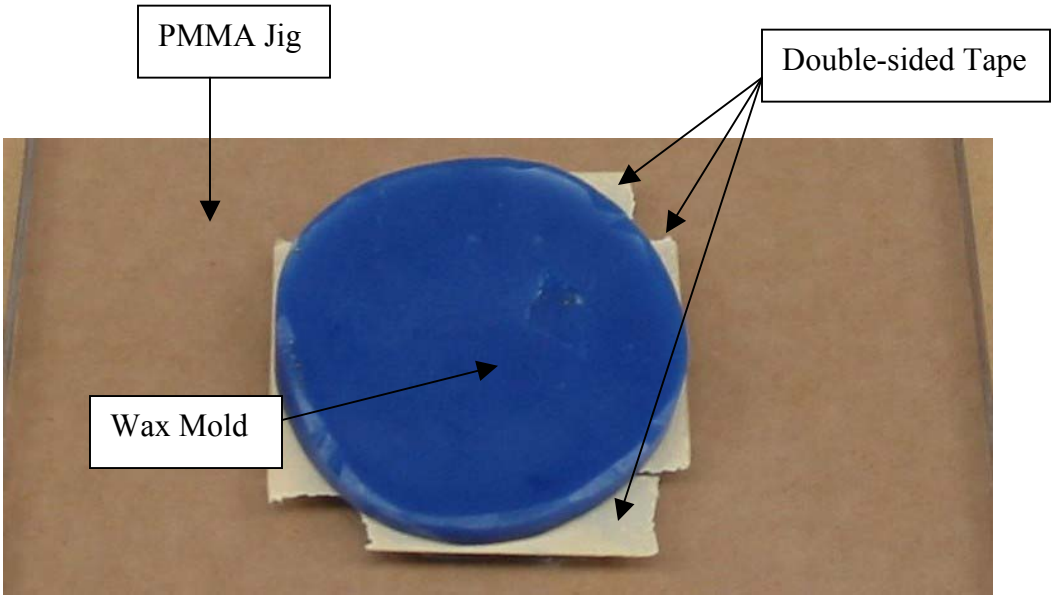

Figure 2-15: Top View of Wax Mold Taped to PMMA Surface

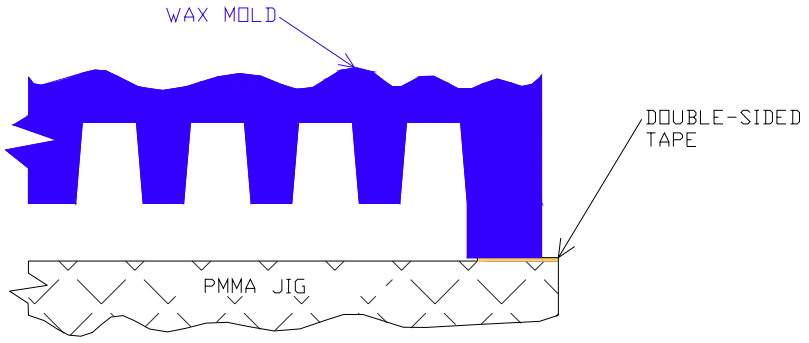

Figure 2-16: Cross-sectional View of Wax Mold Taped to PMMA Surface

After mounting the wax mold, the PMMA jig and wax mold is clamped to the machining surface of a milling machine. A flycutting tool is then used to remove the excess wax material. The flycutting tool's cutting edge had a steep rake angle and was exceptionally sharpened to achieve the optimal machining conditions as recommended by [5]. Despite these optimal conditions, the fly-cutting tool inserted wax into the holes as it removed material surrounding the microholes. Figure 2-17 demonstrates this phenomenon and Figure 2-18 shows the resulting wax template of this process.

Different types of methods were attempted to remove the clogged wax. High-pressured air was blown into the microholes without clearing any of the clogged microholes. Applying concentrated water streams to the microholes was not successful either. With the microholes clogged, nickel electrodeposition could not occur onto the metal substrate. Therefore, the wax template was useless. In order to provide useable wax templates, a method was developed to physically block the removed wax from entering the micro holes. This could be achieved by placing a solid into the micro holes before machining and then removing the solid after machining took place.

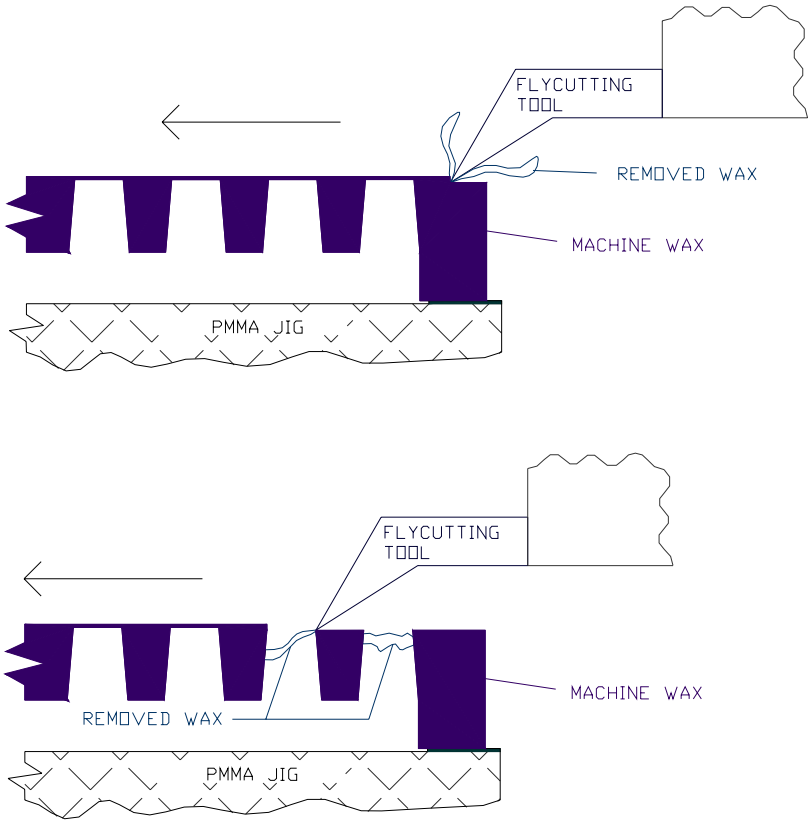

Figure 2-17: Insertion of Wax into the Wax Template Microholes

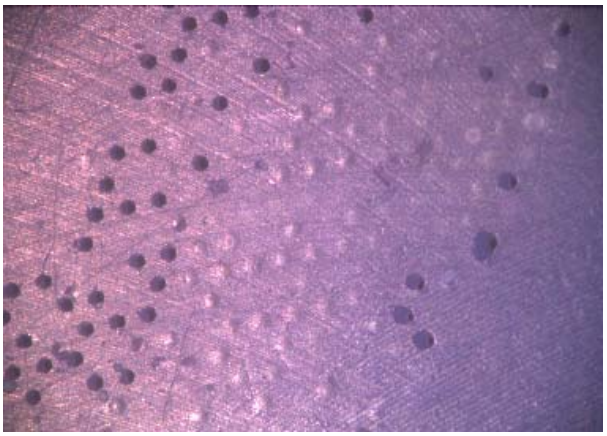

Figure 2-18: Clogged Microholes in Wax Template

NOTE: The opened microholes are black and the clogged microholes are white.

To achieve satisfactory results, three requirements had to be met. First, the solid must be strong and stiff enough to prevent the removed wax from clogging the microholes. Second, the solid must be able to be inserted into the microholes. Third, the solid must be able to be removed from the microholes without damaging the wax template. Satisfying these requirements was the critical step in this template production process. Without the knowledge to solve these requirements, this template production process would not provide a successful template. Without a successful template, the ultimate goal, a mechanical seal bonded with a LIGA heat exchanger, would not be achieved either.

Several combinations of materials, insertion, and removal techniques were investigated to solve these requirements. After several unsuccessful attempts, the IPS Weld-On Acrylic Cement #42® was investigated. The acrylic cement's hardness and strength indicated that it would prevent the removed wax from clogging the microholes. The acrylic cement is typically applied as a gel that cures and hardens. This property indicated that the acrylic cement could be applied to the wax molds as a gel and then immediately placed under vacuum. Vacuuming would remove the trapped air pockets in the microholes and allow the gel to fill the microholes. The gel could then cure and solidify in the microholes. The only requirement left was how to remove the acrylic without damaging the wax template.

To solve the removal issue, two experiments were conducted. The first experiment immersed the machine wax into an acetone bath for twenty-four hours to see how the wax would chemically react. Unlike most waxes, the machinable wax did not suffer any degradation due to the acetone. The second experiment subjected an acrylic

cement to an acetone bath for twenty-four hours. In this experiment, the acetone dissolved the majority of the acrylic cement within ten hours.

These two experiments proved that the acrylic cement could be removed without degradation to the wax template. Armed with this promising knowledge, using the acrylic cement as filler could be investigated further. The acrylic cement was successfully inserted into the wax mold's microholes. The wax mold and acrylic cement were then machined, and the acrylic cement was successfully removed from the machined wax mold (template). The following sections state how these steps were achieved.

2.4. Acrylic Cement Application

The IPS Weld-on Acrylic Cement #42® is a two-part acrylic cement that is shipped in a cartridge containing two cylinders. The cartridge is typically stored in refrigerated conditions to increase its shelf life. Before applying the acrylic cement, the cartridge is removed from the refrigerated conditions and allowed to reach room temperature. Figure 2-19 shows the acrylic cement mounted in its specially designed application gun available for purchase through IPS®. Please note the extended syringe with mixer attached to the acrylic cement cartridge. The purpose of the extended syringe is to completely mix the two-parts of the acrylic cement before application to the desired surface. The acrylic cement is applied to the top of the wax mesh as shown in Figure 2-20.

Once the acrylic cement is applied, the acrylic cement and wax mold is then placed into a vacuum chamber to remove the voids of air trapped in the microholes. A vacuum of 25 in Hg was achieved and then cycled every three minutes over a period of

approximately ten minutes. Vacuuming for a longer period of time led to poor results as shown in Figure 2-21.

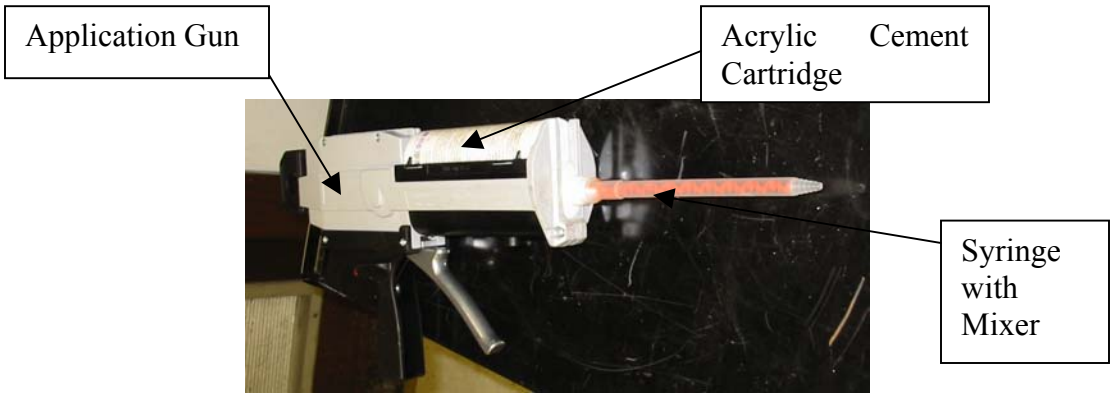

Figure 2-19: Acrylic Cement Applicator

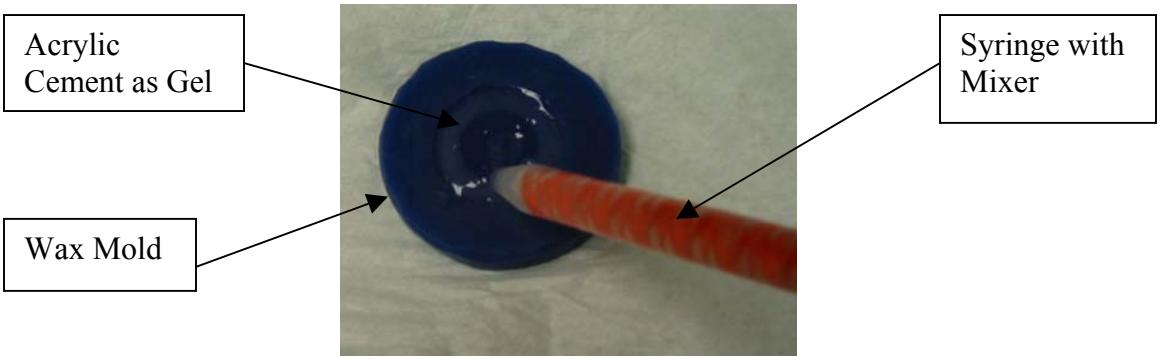

Figure 2-20: Acrylic Cement Application to Wax Mold

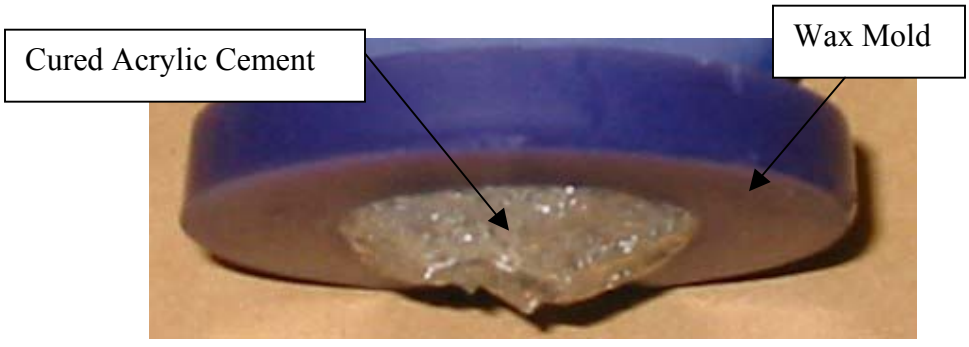

Figure 2-21: Acrylic Cement after Excessive Vacuuming

2.5. Wax Mold With Acrylic Cement Machining

With the acrylic cement present in the microholes, machining the wax mold could occur. However, after applying the acrylic to the wax mesh piece, the acrylic often formed a hemispheric shape during vacuuming that often exceeded the height of the wax template as previously shown in Figure 2-21. This property made the wax mold unmachinable because the wax mold's flat area could not be placed flush with the PMMA jig. The double-sided tape could grab the wax mold's flat surface and hold it stationary under these conditions.

To overcome this obstacle, the PMMA jig was modified to include circular cavities ¼" deep as shown in Figure 2-22. These cavities allow a space for the hemispherical acrylic cement to occupy while also allowing the wax mold's flat surface to be flush with the PMMA jig. The wax template could then be held stationary by the double-sided tape as shown in Figure 2-23.

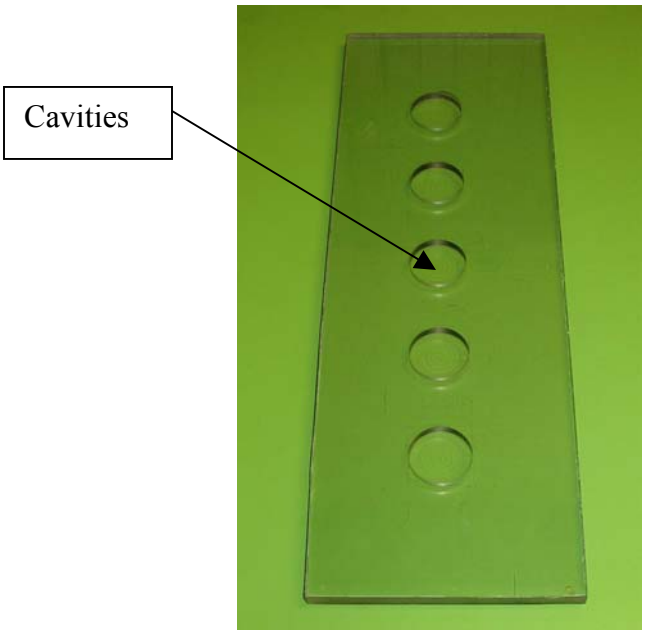

Figure 2-22: Modified PMMA Jig with Cavities

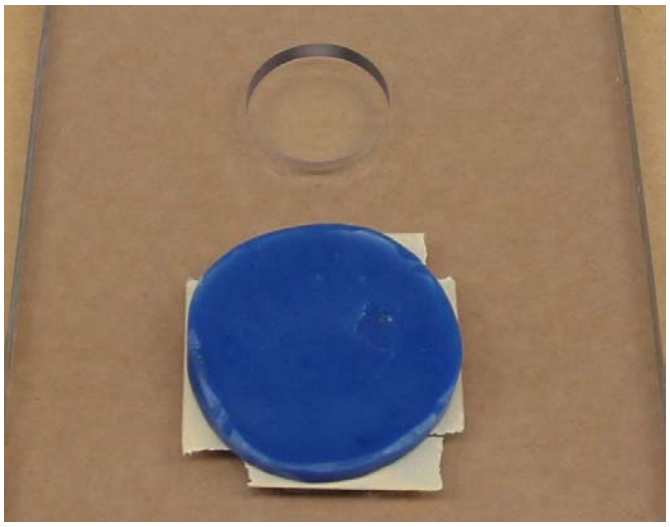

Figure 2-23: Wax Mold with Acrylic Cement Taped to Modified PMMA Jig

The following steps are performed to prepare the surface for the best possible adhesion between the wax mold and double-sided tape. First, the flat area of the wax mold is sanded with 400 grit sandpaper. The sanding smoothed any blemishes that inhibit adhesion to the double-sided tape. Second, a blade is used to remove a thin raised edge along the wax mold's outer diameter edge. This raised edge is due to gaps between the silicone mold and masking tape when the wax pouring occurred.

The machining jig contained five holes to allow the machining of five wax molds at one time. The modified PMMA jig is then mounted to machining surface of the milling machine. The excess wax is then flycut at a spindle rate of approximately thirteen hundred rpms and a feed rate of twenty-four inches per minute.

In order to separate the wax mold from the modified PMMA jig, a piece of metal with thickness of 0.002", called a shim, is used. The shim is gently slid between the double-sided tape and wax template. The shim is patiently applied in this manner over the entire contact area to separate the wax template and double-sided tape. Once the

entire contact area is separated, the wax template is gently removed. Great care is taken with this step to prevent breaking the fragile wax template.

2.6. Acrylic Removal

Before removing the acrylic cement, the boltholes required to clamp the template to the substrate jig are drilled into the wax template. The boltholes are drilled before removing the acrylic cement because the wax template is less fragile when it contains the acrylic cement. Please see section 4.1 on how the boltholes are aligned and drilled.

The wax template with acrylic cement is placed in a 200 ml acetone bath with slight agitation for eighteen hours. The wax template is then placed in a fresh 200 ml acetone bath for four hours with increased agitation to remove any remaining acrylic. The result from dissolving the acetone is shown in Figure 2-24

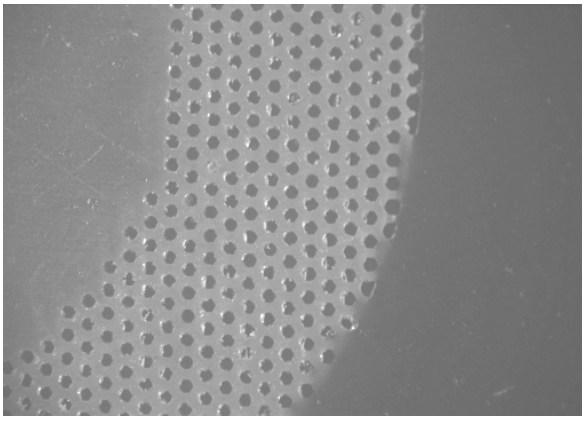

Figure 2-24: Wax after Acetone Baths.

As can be seen in Figure 2-24, the wax mesh is not completely void of burrs, but was vastly improved from previous efforts. The remaining burrs are attributed to the acrylic cement's volumetric shrinking during the curing process. In order to produce quality microposts for the LIGA heat exchanger, the remaining burrs needed to be

removed. To remove the remaining burrs, the wax template is rinsed with streams of DI water, alcohol, and compressed air. The wax template is then examined under a microscope and a micro drill with a diameter of 210 μm, available from MSC® Industrial Supply, Inc., is then used physically remove the burrs.

The result of these refinements can be seen in Figures Figure 2-25 and Figure 2-26.

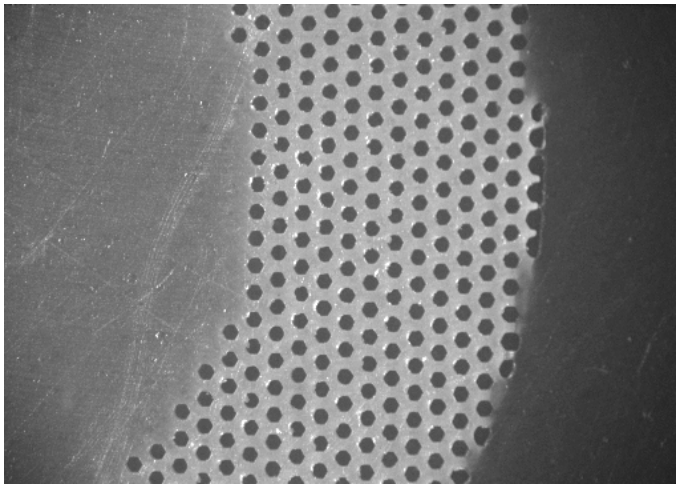

Figure 2-25: Wax Mesh after Further Rinsing.

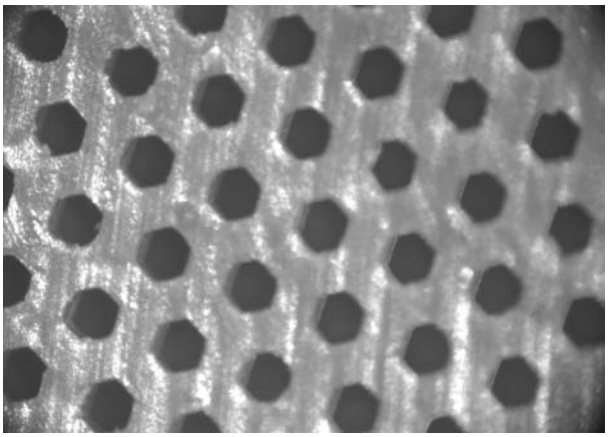

Figure 2-26: Magnified View of Rinsed Wax Mesh

CHAPTER 3. SUBSTRATE PREPARATION

3.1. Substrate Physical Preparation

As stated in the introduction, the bond strength between the nickel micro heat exchanger and mechanical seal needed to be improved. Two methods of attack were pursued to improve the bond strength. One method was to chemically treat the mechanical seal's plating surface. This method is investigated in Sections 3 and 4 of this chapter.

The other method of attack is to physically modify the mechanical seal (or substrate) in order to enhance the conditions for improved bond strength. The most significant physical modification is increasing the plating surface area. Sandblasting the plating surface and allowing unexpected underplating, the electrodeposition of nickel beneath the wax template, achieve this. Besides the benefit of increased bonding area, the sand blasting and underplating provided a foundation with thousands of micro footings for the nickel to become mechanically locked to the substrate.

Another minor physical modification was providing a secure electrical connection for the electroplating process. This requires a sound solder connection between an electrical wire and substrate. The following steps were taken to achieve this. A hole is drilled into the substrate on the same side as alignment holes to hold solder as shown in Figure 3-1

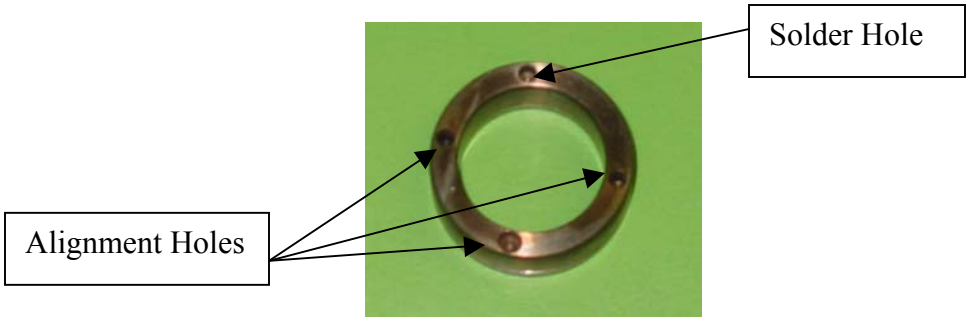

Figure 3-1: Drilled Hole for Solder in Substrate

The newly drilled hole is then rinsed with soap, de-ionized (DI) water, and alcohol. A silver, tin, copper alloy solder is placed into the solder hole. The substrate and solder are then heated by a hot plate to melt the solder.

An insulated electrical wire of gauge eighteen and three hundred volts rating is used for this application. The insulated electrical wire is cut to a length of approximately three feet long. Approximately one inch of the wire's insulating material is removed at one end. The bare wire at this end is cleaned with alcohol and DI water. The bare wire is then placed onto the hot plate for approximately one minute to increase the wire's temperature. The hot bare wire is then immersed into the solder hole and liquid solder. The substrate and electrical wire are simultaneously removed from the hot plate. The electrical wire is held in the solder hole while the solder is allowed to solidify.

The second physical modification made to the substrate was sandblasting the substrate's plating surface and therefore, increasing the surface area. The substrate is placed into a typical manual sandblasting machine. The substrate's plating surface is evenly sandblasted until the plating surface was uniform. Figure 3-2 illustrates the difference in substrate's plating surface before and after sandblasting.

The result of the sand blasting the surface and clamping the template to this surface is spatial voids between the surface and template. During the electroplating process, these voids are filled with electroplated nickel and result in the previously mentioned underplated foundation of approximately 60 microns. Figure 3-3 shows the underplated nickel layer and electroforming of the heat exchanger's nickel micro posts.

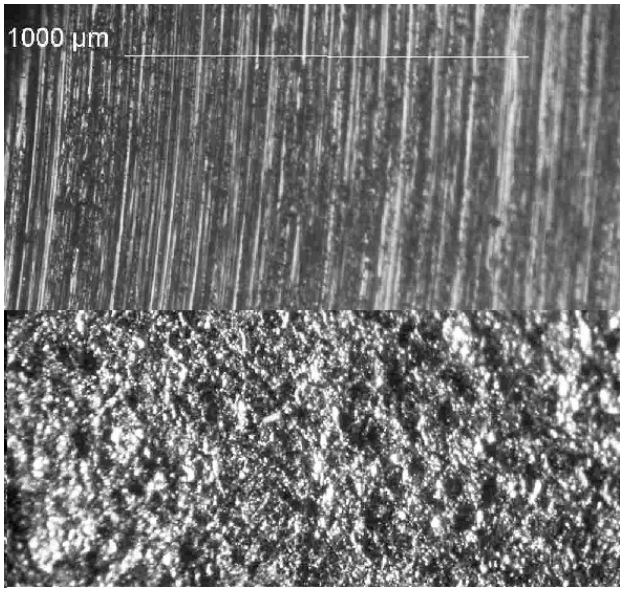

Figure 3-2: Surface before Sandblasting (Top) and Sandblasted Surface (Bottom)

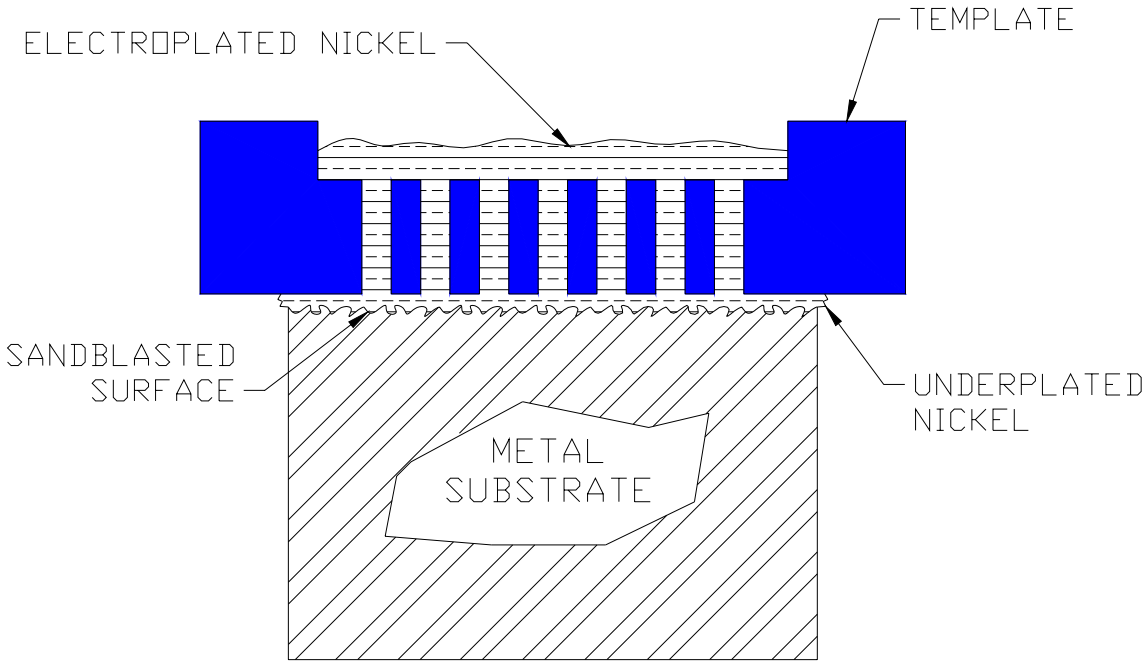

Figure 3-3: Cross Sectional View of Underplated Nickel and Sandblasted Surface

The underplated nickel layer was not originally planned and discovered after the first sample was electroplated. Underplating did offset the seal's heat exchanger from the seal's plating surface by approximately 60 μm. For this application, this alteration is considered minor and worth the advantage of additional bonding area. Without the underplated nickel layer, the LIGA heat exchanger would only be bonded to the mechanical seal at the base of the micro posts. The underplated nickel layer existence allows bonding over the entire surface area of the metal substrate. This increased the surface area by a factor of two. Figure 3-4 shows the actual underplated nickel layer. Please note the inner and outer diameter irregularities. These irregularities required additional machining that is stated in Chapter 5 Section 4.

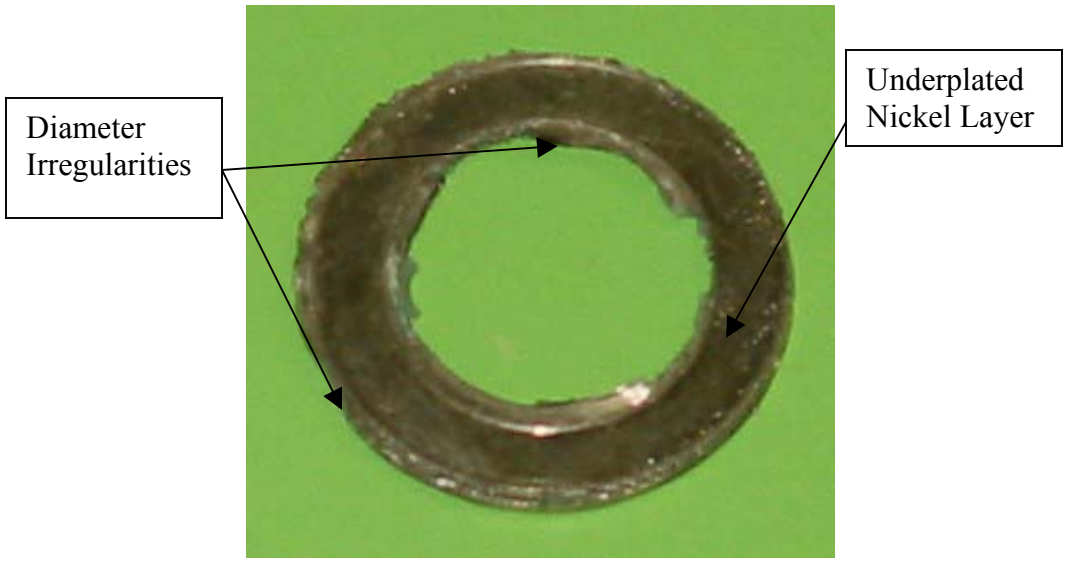

Figure 3-4: Underplated Nickel Formed During Electroplating

3.2. Substrate Jig Assembly

For substrate to be chemically treated, the substrate jig described in Chapter 1 Section 3 is used and supplied by the University of Kentucky. Figure 3-5 shows an isometric and

exploded view of the substrate jig. Figure 3-6 shows a cross-sectional view of the substrate jig assembled.

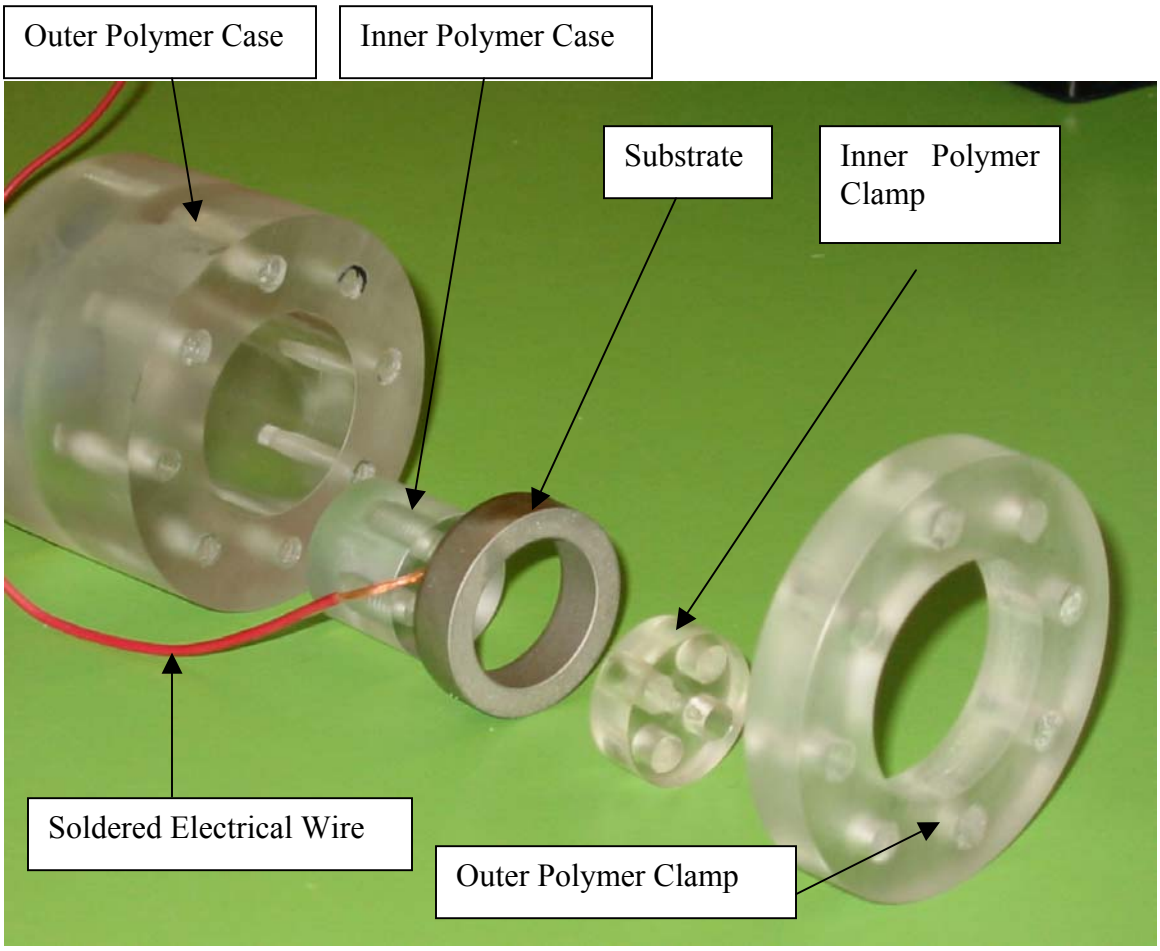

Figure 3-5: Isometric and Exploded View of Substrate Jig

In order to assemble the substrate jig, the following steps are taken. The inner polymer case is placed into a freezer and subjected temperatures lower than 0°C for approximately two hours. The substrate is then placed onto a horizontally flat surface with the alignment holes facing upwards. The inner polymer case is then pressed into the substrate's inner diameter with the inner polymer case threaded boltholes facing

downwards. The inner polymer case and substrate is allowed to reach room temperature in the configuration shown in Figure 3-7.

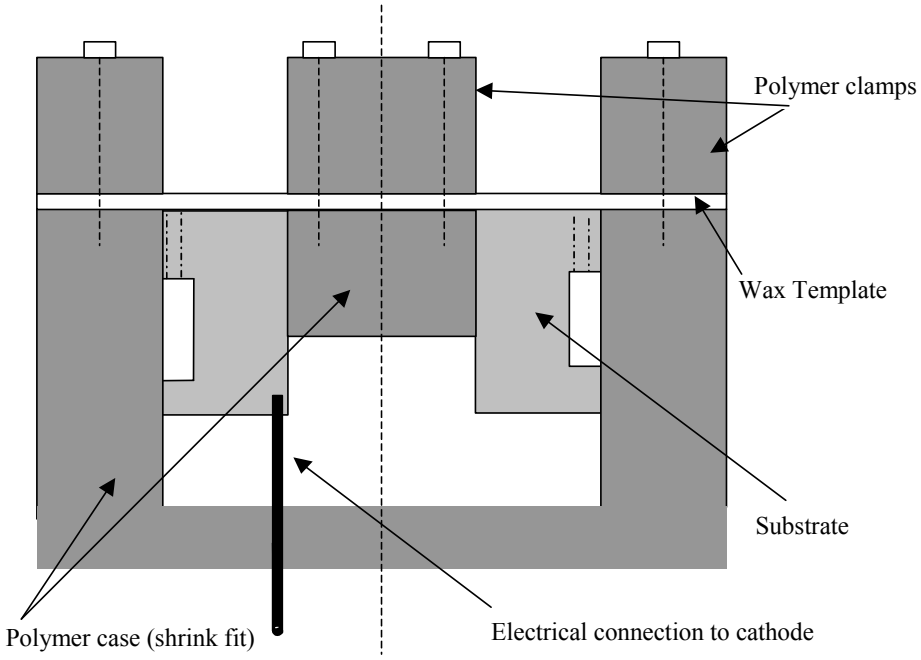

Figure 3-6: Cross Sectional View of Substrate Jig, Modified Version from [1]

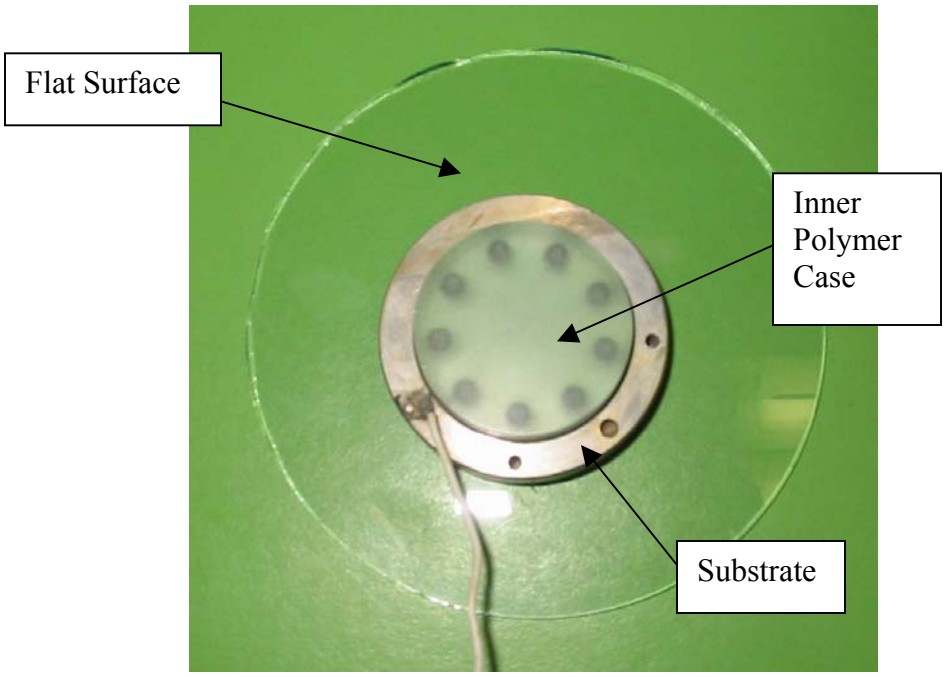

Figure 3-7: Inner Polymer Case Pressed into Substrate's Inner Diameter

The outer polymer case is placed into an oven at 80°C for twenty minutes. The outer polymer case is removed from the oven and the electrical wire is pulled through a 1/16" through hole located at the base of the outer polymer case. Figure 3-8 demonstrates the wire being passed through the hole.

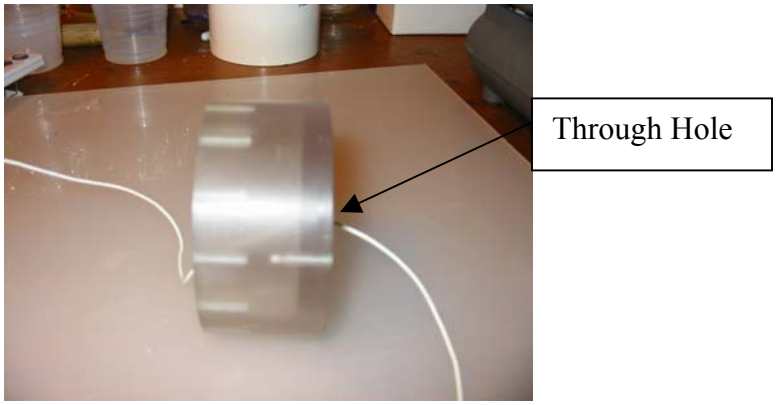

Figure 3-8: Electrical Wire Pass Through the Outer Polymer Case Through Hole

The heated outer polymer case is then pressed onto the substrate and inner polymer case. All three pieces are pressed against the same flat surface until the lower base is flush against the flat surface. The pieces are then allowed to reach room temperature in the configuration shown in Figure 3-9.

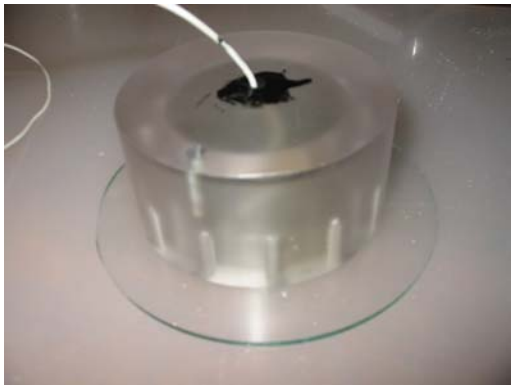

Figure 3-9: Outer Polymer Case Pressed onto Substrate and Allowed to Cool

North American Brush-On Electrical Tape® is then applied generously to the outer polymer case through hole from which the electrical wire is allowed to pass. The brush-on electrical tape seals the gap between the hole and the electrical wire running through the hole. The liquid electrical tape is allowed to cure for four hours as shown in Figure 3-10.

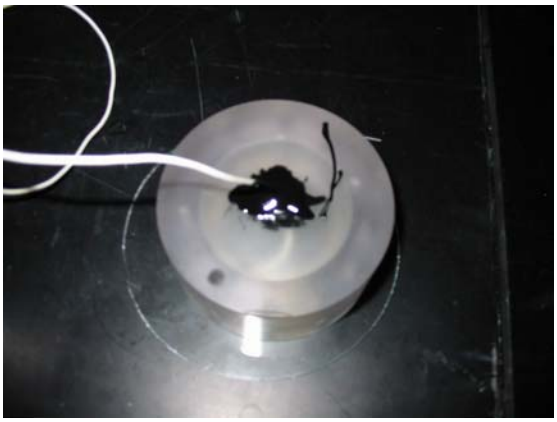

Figure 3-10: Brush-on Electrical Tape Curing

3.3. Activation Procedure

An activation process is needed to establish a strong bond between the electroplated material and the stainless steel substrate. Activation removes an adherent, non-porous, passivating oxide film that forms on the substrate. [12] This oxide film prevents strong bonds between the electroplated nickel and the substrate. Once the oxide layer is removed, the electrodeposited nickel bonds directly to the substrate and results in a stronger bond. The activation process is as follows:

1. **Six liters of activation solution is prepared to activate the substrate.**

The activator solution prepared consisted of the composition shown in Table 3-1. Concentrated C-12 Activator was used as the activating agent and is available from Puma Chemical. [9] Diluted sulfuric acid is added to the solution under heavy agitation until the pH of the solution reaches 1.5. The solution is then filtered before each use with a filter of porosity equal to 1.6 μm.

Table 3-1: Composition of Activator

Compound	Concentration (% Volume)
De-ionized Water	97.5
Concentrated C-12 Activator	2.5
Sulfuric Acid (H_2SO_4)	<1, Sets pH=1.5

2. A plating jig was fabricated to hold the substrate jig in the activator, wood strike, and electroplating solutions and shown in Figure 3-11.

This plating jig consisted of two square 3" x 3" pieces of PMMA with a thickness ½". The upper PMMA piece was machined to have an angled bottom. The lower PMMA piece was then glued to the angled bottom with Loctite 415. This produced a plating jig that held the substrate's surface at an angle. Electroplating the substrate at an angle proved to be critical for reasons discussed in Chapter 4 Section 3.

A through hole of diameter of 0.2125" was cut into the angled plane of the jig lower to hold the substrate jig with a nylon screw. Two threaded holes were machined on the top plane to allow the plating jig to be bolted to the tank support jig as shown in Figure 3-12. Nylon bolts were used in each chemical process to

eliminate corrosion and electroplating problems associated with the use of metallic bolts. An arm was glued to the jig to facilitate the handling of the jig.

Figure 3-11: Plating Jig for the Activation, Wood's Strike, and Electroplating Processes

3. The substrate jig is then bolted to the plating jig by using nylon bolts.

Note: The polymer clamps and wax templates are not used during this C-12 activation process or the Wood's strike process.

The polymer clamps and wax templates are not used because they increase the difficulty of activating and Wood's striking the surface. The two jigs are then bolted to a tank support jig made of PMMA. The tank support jig holds the plating and substrate jig in the activation solution as shown in Figure 3-12.

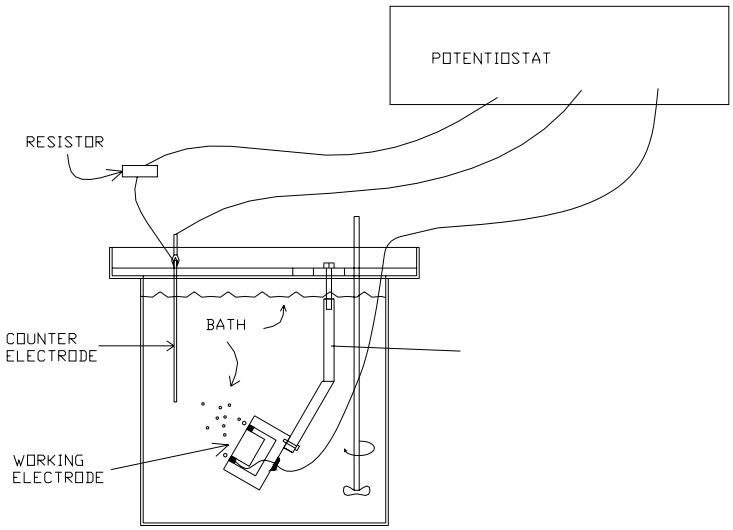

Figure 3-12: Activator Bath Setup

4. A constant voltage of negative two volts was applied between the counter electrode and substrate for one and a half minutes.

The electrical layout required to achieve this is shown in Figure 3-13 and is wired to the AMEL® potentiostat as recommended by the manufacturer. A piece of stainless steel with dimensions of 8" x 4" with a thickness of .025" is cleaned and partially placed into the activator solution. The stainless steel anode is connected to the potentiostat's counter electrode connection by using an insulated electrical wire and alligator clip. A 1,000 Ω resistor is connected in parallel to the reference electrode and stainless steel anode. The manufacturer recommends the including the resistor. The purpose of the resistor is to limit the current applied to the reference electrode and simultaneously maintain a constant voltage between the counter electrode and working electrode [10].

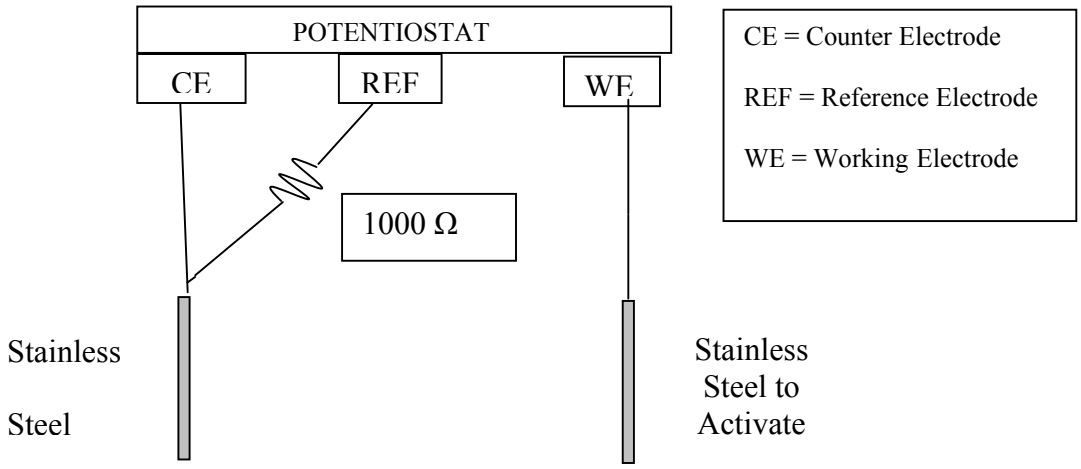

Figure 3-14: Electrical Setup of Activating Process

The substrate's insulated electrical wire is then connected to the working electrode connection on the potentiostat. All of the electrical connections are then

tested with an ohmmeter to verify electrical continuity. A constant voltage of negative two volts is then applied between the stainless steel anode and stainless steel substrate. The constant voltage is applied for one and a half minutes while the applying strong agitation to the solution.

The activation reaction produces gas bubbles on the surface being activated. Some bubbles became trapped and remained on or near the activating surface despite applying rigorous bath agitation. With the presence of these bubbles near the surface, the surface is not activated, i.e. the thin oxide layer is not removed. The result is a poorly bonded nickel and weak interface between the mechanical seal and LIGA heat exchanger. To overcome this obstacle, the plating jig and substrate jig are manually shaken vigorously every fifteen seconds during the activation process. The result is the release of trapped bubbles and the surface being activated for a longer time period.

5. The substrate is removed from the activation bath.

After activating the surface, it must not be exposed to air. The air exposure re-forms the thin oxide layer. Therefore, the plating jig and substrate jig are removed from the activator solution in the following manner:

i) The plating jig is unbolted from the tank support structure and the entire jig is rotated such that the substrate orientation is changed from vertical to horizontal with the substrate facing upwards. Throughout the process, the substrate is submerged in the activator solution. The jig is then removed carefully, maintaining the horizontal orientation of the substrate. In the horizontal orientation, surface tension provides a thin a thin layer of

activation solution to remain above the activated surface. Therefore, this procedure prevents the activated surface from being exposed to air as the substrate is moved from one plating bath to another.

ii) In order to minimize the activation solution from entering baths used later, the jig is rinsed in de-ionized water while supported over the activator solution. The de-ionized water is gently sprayed so that no region is exposed to air. The jigs are then placed into the Wood's strike bath and bolted to the tank support jig in the same orientation as shown in Figure 3-12.

3.4. Wood's Strike Procedure

The purpose of the Wood's strike procedure is to apply a four-micron layer of nickel onto the stainless steel substrate [11] through an electrolytic cell. The thin nickel layer is applied in a strongly acidic nickel solution. This condition results in the applied nickel being very active and improving the adhesion of subsequent electrodepositions. [12] Due to efficiency issues, this process is only feasible for applying thin layers of nickel. However, the four-micron nickel layer serves as a buffer that is well-adhered to the substrate and that allows the electrodeposited nickel to bond to nickel. Bonding similar metals has been shown to improve bonding strength and promote epitaxial growth, the electrodeposition of a metal such that the atomic arrangement of the crystalline substrate is perpetuated during the electrodeposition. [8]

The Wood's strike process is as follows:

1. Six liters of Wood's strike solution is prepared.

The Wood's strike solution prepared consisted of the composition shown in Table 3-2. It is important to note that the nickel chloride and four liters of DI were mixed first. The hydrochloric acid was then added. The remaining volume was filled with DI water. The solution is then filtered before each use with a filter of porosity equal to 1.6 μm

Table 3-2: Composition of Wood's Strike Bath

Compound	Formula	Concentration (g/l)
Nickel Chloride (metal basis)	$NiCl_2 \cdot 6H_2O$	240
Hydrochloric acid	HCl	80

2. A constant current density of fifty milliamps per square centimeter of plating area is applied to the substrate's surface for seven minutes.

The electrical layout required to achieve this is shown in Figure 3-14. A nickel anode is required for this process. A nickel foil of 99.5% (metals basis) of dimensions 100 mm x 100 mm x 0.787 mm thick is used for the anode. The nickel foil is partially held into the Wood's strike solution in a similar fashion as the stainless steel anode shown in Figure 3-12. The anode and substrate are physically connected to the potentiostat in the same manner as stated in the activation process. An ohmmeter is then used to verify the electrical continuity.

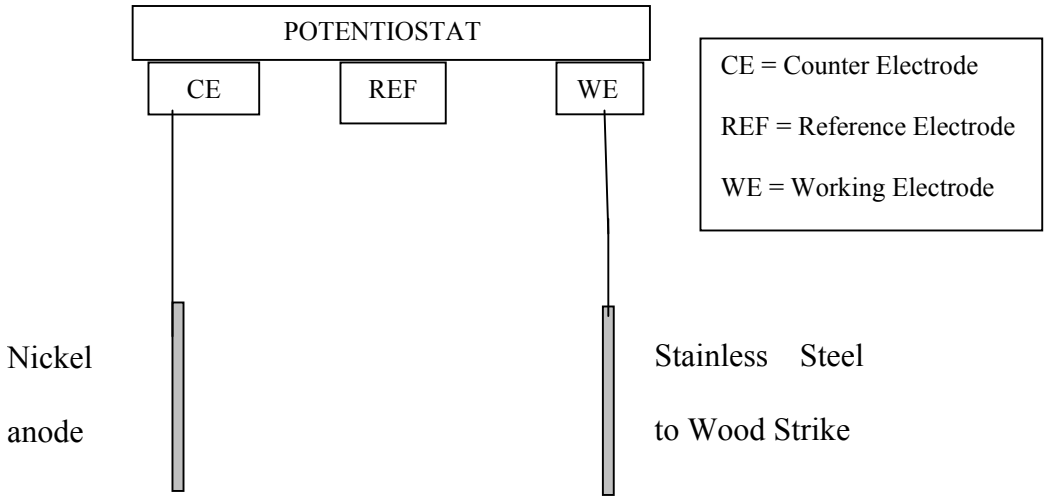

Figure 3-14: Potentiostat Electrical Setup for Wood's Strike Process

The potentiostat imposes a constant current density of fifty milliamps per square centimeter of plating area. The total constant current supplied to the plating surface for this application is one hundred and eighty-eight milliamps. This current is applied to the surface for seven minutes.

The Wood's striking chemical process also forms gas bubbles at the plating surface. Strong agitation of the solution and buoyancy forces helps remove most of the gas. To aid in the removal of the remaining bubbles, the plating jig and substrate jig are manually shaken. The removal of the gas ensures a uniformly plated layer of nickel. The advantage of a uniform nickel layer is that it leads to stronger bond strength between the nickel posts of the LIGA heat exchanger and stainless steel substrate. The stronger bond at this interface provides a stronger mechanical seal. The plating jig and substrate are then

unbolted from the tank support jig and removed from the Wood's strike bath. The plating and substrate jigs are rinsed with DI water over the Wood's strike bath and allowed to dry.

CHAPTER 4. ELECTRODEPOSITION

4.1. Wax Template and Substrate Jig Assembly

Once the plating surface and wax template is prepared as stated in Chapters 2 and 3, the wax template and substrate jig is assembled for the electrodeposition process. The first step of this assembly requires through holes to be drilled into the wax template. The wax through holes are required to bolt the substrate jig's polymer clamps to its polymer cases and consequently, clamp the wax template to the substrate.

Drilling the through holes into the wax template is slightly problematic because the bolthole geometry of each substrate jig varies. The source of this variation occurs due to the press fit of the polymer cases. The inner and outer polymer cases have a fixed threaded bolthole orientation. However, when the polymer cases are press fit along the substrate, the relative angle between the inner and outer polymer cases' threaded boltholes varies. Without a constant thread hole geometry, the wax template holes have to be drilled after press fitting the polymer cases.

To solve this problem, the outer polymer clamp with the predrilled holes is taped onto the top of the wax template as shown in Figure 4-1. This provides a template to drill the outer boltholes into the wax template. The boltholes are first drilled with a 3/16" bit. A 7/32" bit is then used to increase the through hole diameter.

Next, the wax template's inner through holes are drilled. In order to complete this step, the substrate jig and plating jig are removed from the Wood strike bath and rinsed with DI water. The outer polymer clamp and wax template are then taped and bolted to the substrate jig as shown in Figure 4-2. The previously drilled bolt holes of the wax template and outer polymer case bolt holes are aligned before taping the pieces together

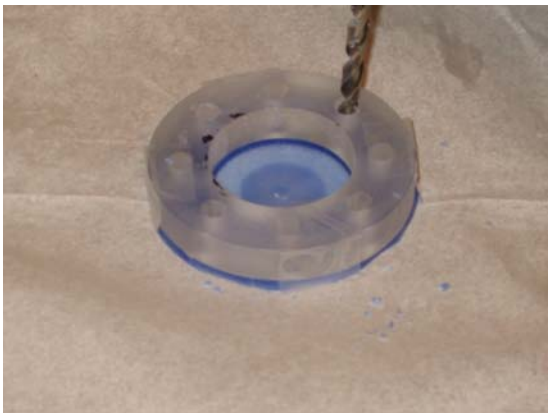

Figure 4-1: Drilling the Outer Wax Template Through Holes

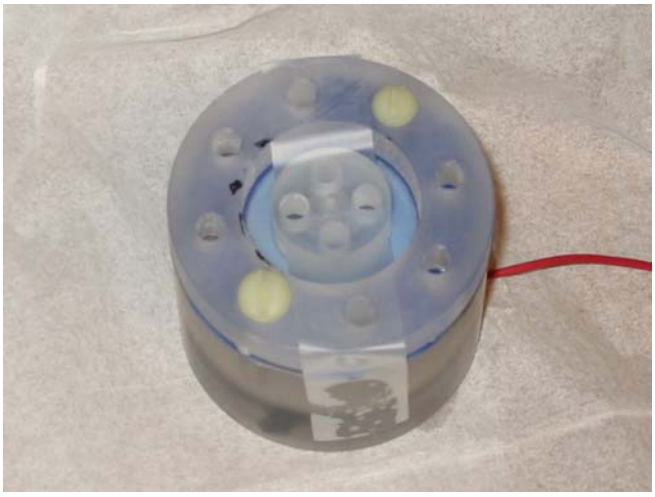

Figure 4-2: Inner Polymer Clamp, Outer Polymer Clamp, and Wax Template Taped to Polymer Case

As also shown in Figure 4-2, the inner polymer clamp is taped to the substrate jig. In order to tape the inner polymer clamp in the proper orientation, the following steps were taken. The wax template and outer polymer clamp are clamped together *without* the inner polymer clamp. The threaded boltholes of the inner polymer case are seen through wax template due to wax template's opaqueness. A black mark is applied to the wax

template at the bolthole locations. The inner polymer clamp is then oriented so that the clamp's through holes and marks are aligned. The inner polymer clamp is then taped to the substrate jig as shown in Figure 4-2 to provide additional stiffness to the wax template during the drilling of the through holes.

Due to the wax template's thickness of less than 1 mm, the wax template tended to crack easily when drilling. The cracked templates were thrown away. To minimize the possibility of cracking, a drill bit of diameter 3/32" is first used to drill the holes because a smaller drill diameter minimizes the torque required to drill the hole. A drill bit of diameters 1/8" and 3/16" are then successively used to enlarge the through hole. The inner polymer clamp, outer polymer clamp, and wax template are carefully separated and cleaned to remove any loose wax. The wax template is then immersed into an acetone bath to remove the acrylic cement as previously stated in Chapter 2 Section 6. The final result of the wax template is shown in Figure 4-3.

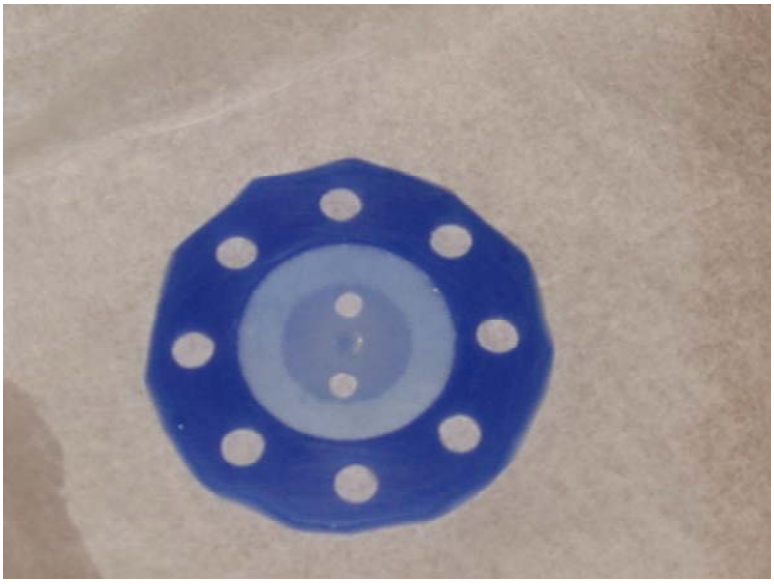

Figure 4-3: Wax Template after All Modifications

The wax template is then placed back onto the substrate jig with the boltholes aligned. The outer polymer clamp, inner polymer clamp, and the wax template are bolted to the inner and outer polymer cases with nylon bolts.

4.2. Re-Activation Process

Once the substrate is removed from the Wood strike solution, the thin oxide later tends to re-form and prevent strong bonds to the newly electrodeposited nickel. Therefore, the plating surface needed to be activated once again. In the first activation process, the sample is placed directly into the solution and shaken vigorously during the activation. This procedure is slightly modified because of the wax template and polymer clamps are now part of the substrate jig. The first adjustment is the filling the channel created by the polymer clamps and wax template with a solution of lauryl sulfate and vacuuming before activating. The second adjustment is increasing the activation time to two minute time periods, vacuuming, and activating again.

The reason for adding the lauryl sulfate solution before activating is that air becomes trapped in the micro holes of the wax template when the substrate jig is submerged into the activator solution. The presence of these bubbles prevents the activation of the plating surface and could not be tolerated. To correct this problem the following procedure is followed. A solution is made consisting of three grams of lauryl sulfate and one liter of de-ionized water. This solution possesses relatively low surface tension forces and allows the lauryl sulfate solution to enter the micro holes during vacuuming. The substrate jig is held so that the plating surface is horizontal as the lauryl sulfate solution is poured into the substrate channel. The substrate and lauryl sulfate are then placed in a vacuum chamber. A vacuum of 28.5 in Hg is applied for approximately

ten minutes and then released. This vacuum cycle is repeated until all of the trapped air is removed.

The substrate jig and lauryl solution are then held horizontally as the substrate jig is completely submerged into the activating solution. The activation procedure stated in Chapter 3 Section 3 is then performed. During this activation, the time length of activation is two minutes. The plating jig was shaken even more vigorously. Despite the time length and additional shaking, some gas bubbles were still trapped. To remove the gas, the substrate jig is removed horizontally from the activating solution and the vacuum cycle is repeated. The substrate jig is then horizontally placed back into the activating solution and the activation is repeated. The cycle of vacuuming and activating is performed at least two times for each mechanical seal. The substrate jig and plating jig are left submerged in the activator solution while the nickel electroplating solution is prepared for electroplating. The substrate and plating jig are then removed from the activator solution and placed into the electroplating solution in the same manner to prevent the substrate surface from being exposed to air.

4.3. Nickel Electroplating Process

The purpose of the nickel electroplating process is to efficiently electrodeposit significant amounts of nickel onto the substrate in the opposite pattern of the wax template. The nickel electroplating process is as follows.

1. The electroplating solution is prepared.

The six-liter electroplating solution prepared was a nickel sulfamate bath consisting of the composition shown in Table 4-1. The tank containing the solution is placed in an isothermal water tank that maintains the electroplating

solution temperature at 55˚C. A nylon stirrer then applies heavy agitation to the solution. The solution is undisturbed for twelve hours to allow the solution to completely mix and reach 55˚C. Diluted sulfuric acid is then added to the solution until the solution's pH equals 3.9. The solution is then filtered before each use with a filter of porosity equal to 1.6 μm.

Table 4-1: Composition of Nickel Sulfamate Bath

Compound	Formula	Concentration (g/l)
Nickel Sulfamate	$Ni(SO_3NH_2)_2 \cdot 4H_2O$	450
Boric Acid	H_3BO_3	37.5
Lauryl Sulfate, Sodium Salt	$C_{12}H_{25}O_4SNa$	3

2. **The plating and substrate jigs are removed from the activation bath with the plating surface horizontal.**

This results in activator solution filling in the void between the top inner cylinder and top outer ring. DI water is then sprayed generously onto both jigs and void in order to dilute any activator solution that enters the electroplating solution. While the activator solution is stated not to affect the electroplating bath [9], the dilution step is performed to minimize the potential problems of the activator solution in the electroplating solution. It is important to note that the substrate surface is not exposed to air in this step as well steps 3 and 4.

3. **The plating and substrate jigs undergo the vacuum process to remove any bubbles formed during the last activation cycle.**

4. **The substrate jig and plating jig are removed from the vacuum chamber and immersed in the nickel sulfamate bath in the same manner previously stated.**

The plating jig and consequently, the substrate jig are then bolted to the electroplating bath's tank support jig.

5. **Current is applied to applied to the substrate's surface to begin electrodeposition.**

The electrical setup required to achieve this is the same as the Wood strike process and is shown in Figure 4-4.

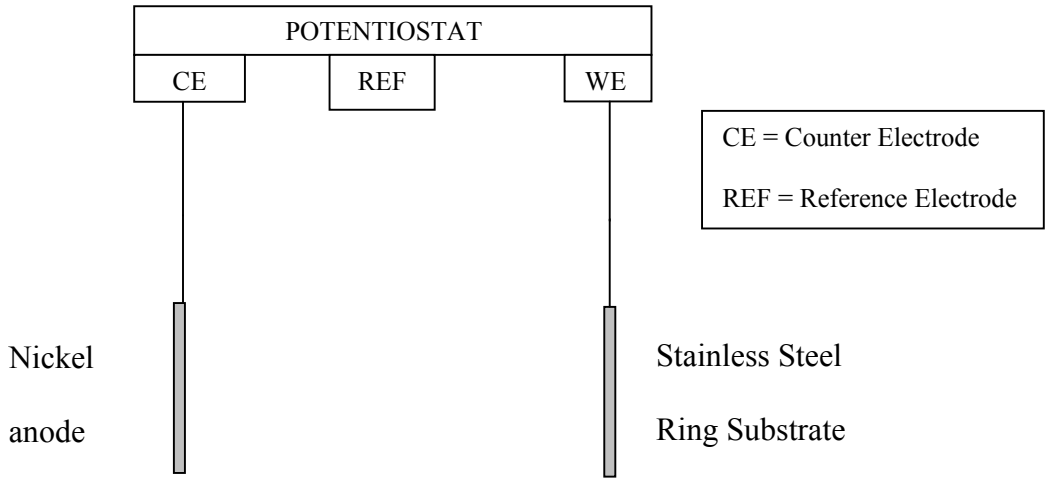

Figure 4-4: Electroplating Electrical Setup

The nickel anode shown above consists of approximately thirty nickel rounds, cylindrical pieces of nickel approximately one inch in diameter and 3/16" thick. The nickel rounds are cleaned with DI water, acetone, and alcohol. The nickel rounds are also left overnight in a diluted sulfuric acid solution containing a volumetric concentration of 3% sulfuric acid. This diluted sulfuric acid helps remove any possible pollutants on the surface of the nickel rounds. The nickel rounds are then placed into a wire mesh bag. The wire mesh bag is made of

titanium and contains a cloth filter to prevent breakdown particles from depositing in the solution. The solution is then filtered before each use with a filter of porosity equal to 1.6 μm. The titanium mesh containing the nickel rounds are placed in soapy water and shaken until all of the breakdown particles are removed from the titanium mesh.

The electrical connections are connected in the same fashion as stated in the Wood strike procedure. A current density of 10 mA/cm^2 is used at first to deposit nickel within the wax template features. A 10 mA/cm^2 current density is considered relatively low because typical current densities for Nickel Sulphamate baths range from 20 mA/cm^2 to 80 mA/cm^2 [8]. This current density is selected because a lower current density is known to provide greater bond strength [13].

The current density determines the rate at which the free nickel ions in the electrolyte solution are deposited on to the substrate. Therefore, the higher the current density, the faster an electroplating height is achieved. Lower current densities permit the slower migration of free ions to the substrate. This increases the opportunity for the ions to be incorporated into the existing lattice structure and favors epitaxial growth [8]. This results in finer grain sizes and better adhesion to the existing nickel layer plated on the substrate during the Wood strike process.

In order to apply a current density, a total current that is based on the substrate's surface area is applied to the substrate surface. The total constant current applied to the substrate is calculated from the following formula:

$$I = J * A$$

Equation 4-1

Where I = the total applied constant current, mA

J = the current density, mA/cm^2

A = the total plated area, cm^2

The total plated area in this case is the area of the stainless sting ring exposed to the plating solution. This area is equal to total area of micro holes. A ratio of the micro hole's area to the unit square area was formulated. The micro hole diameter was measured as well as the distance from center spacing, L, by using a microscope and micrometer. The micro hole was calculated and the unit square area was set equal to L^2. The resulting ratio provided a percentage of how much surface was exposed to the plating solution over the entire substrate surface. The two ratios were then related and are stated below.

$$\frac{A_p}{A_s} = \frac{Area_{microhole}}{Unit\_Square\_Area}$$

Equation 4-2

where A_p = the area to be plated

A_s = the surface area of the stainless steel ring

$Area_{microhole}$ = the open area of one microhole

Unit_Square_Area = the unit area of one microhole and surrounding wax

The only unknown was the total plating area and therefore could be solved. With the total plating area known the total current was calculated and applied to the substrate.

6. The current is increased to provide a current density of 10 mA/cm^2 once the micro holes are filled and the overplated nickel connects.

The current density is then increased 10 mA/cm^2 every twenty-four hours until a current density of 50 mA/cm^2 is achieved.

7. The sample is overplated until a theoretical height of six thousand microns is achieved.

The required overplated surface needed to be flat and have a height of three thousand microns. Due to the nature of electroplating, there are certain areas of the surface that are electroplated unevenly. The result of uneven electrodeposition is hemispherical shapes on the surface and raised edges along the inner and outer polymer clamps. Further machining is performed on the surface to provide a flat surface and state in Section 4 of this chapter. However, the additional machining, uneven electrodeposition, and electrodeposition efficiencies require an additional amount of overplating.

To calculate the time needed to reach a theoretical overplated height of 6000 μm, the following form of Faraday's Law from [7] is used to determine the overplated height.

$$t = \frac{h * \rho_{Ni} * F * n_{elec}}{J * M}$$

Equation 4-3

Where:

t = Time (sec)

h = Plating Thickness (cm)

ρ_{ni} = Density of Nickel (g/cm3)

F = Faraday's Constant (C/mold)

n_{elec} = No. of Electrons Transferred (mol/Equiv)

J = Current Density (Amps/cm2)

M = Molecular weight

The height desired is 0.6 cm, and the density of the nickel plated is 8.9 g/cm^3. Faraday's constant is 96488 C/mol. The number of transferred electrons equals 2 mol/Equiv and the current density is varied from 0.01 $Amps/cm^2$ to 0.05 $Amps/cm^2$. Each time the current density was increased, the height added at that current density was calculated based on the time period. A running total of height was recorded as well. Once a current density of 0.05 $Amps/cm^2$ was achieved, the difference in the recorded height and desire heights was calculated. That difference was then used to calculate the time required to reach the desired height. The time required for the overplating to end was approximately one and a half weeks.

4.4. Electrodeposition Discoveries

After the first sample was produced, a thin solid layer of underplated nickel was observed. This underplating directly affected the current density actually applied to the substrate surface. Instead of applying 10 mA/cm^2 to the substrate, the current density applied was 5 mA/cm^2. A lower current density provided a stronger and successful bond due to a stronger chemical bond and larger bonding surface area. Therefore, the original total current was applied to the samples that followed.

Another discovery was the need for the surface of the substrate jig to be held at an angle. The results after the first attempts of electroplating were not successful due to the

original plating jig. The original plating jig did not hold the substrate jig at an angle. The result of this is shown in the Figure 4-5. Figure 4-5 shows that the top of portion of the seal that did not electroplate at the same rate as the remaining area of the seal. This phenomenon was noted to only take place at the top of the substrate.

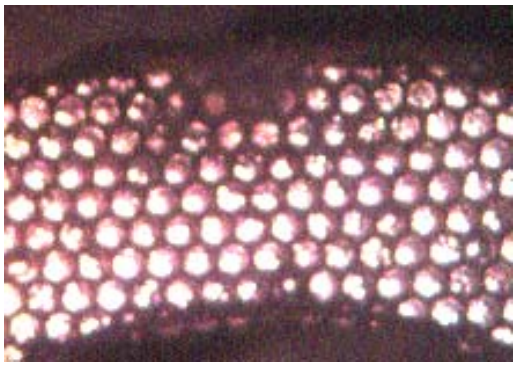

Figure 4-5: Poorly Electroplated Area at Top of Substrate

The reason for this result was found to be due to the electroplating chemical reactions. There were two competing reactions occurring on the cathode, i.e. substrate. [14] These reactions were the following:

$$Ni^{2+} + 2e^{-} \rightarrow Ni$$

$$2H^{2+} + 2e^{-} \rightarrow H_2$$

Equation 4-4

Because the complete nickel evolution does not occur, hydrogen gas forms [14]. The hydrogen gas's buoyancy caused it to rise vertically. The hydrogen gas rose until the outer polymer clamp eventually trapped the hydrogen as shown in Figure 4-6 and Figure 4-7.

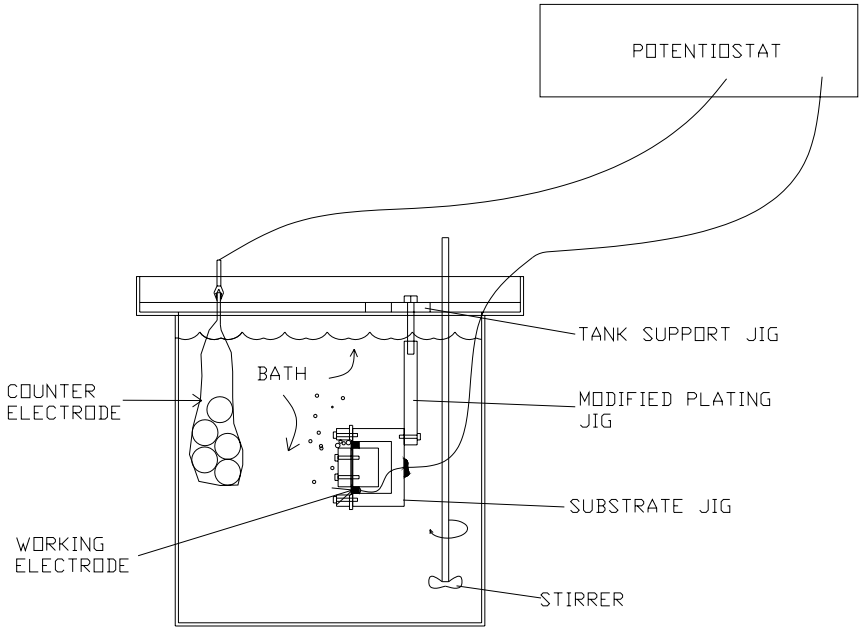

Figure 4-6: Electroplating Bath Setup with Hydrogen Gas Trapped

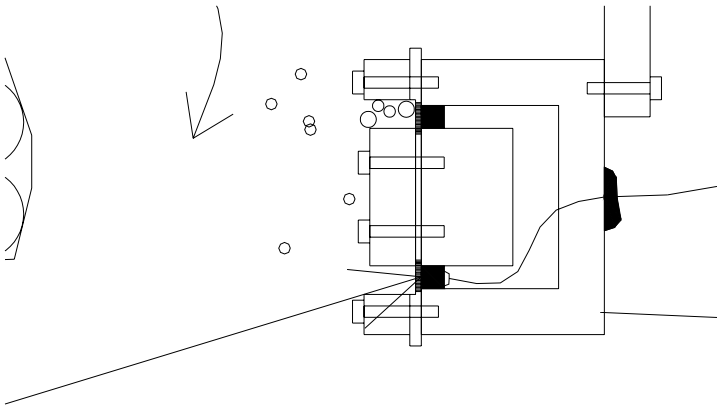

Figure 4-7: Magnified View of Hydrogen Gas Trapped

The result of having a high concentration of hydrogen gas is that the electroplating solution and nickel ions are not able to reach the surface consistently. In

addition, the nickel that is deposited in this area traps hydrogen into the nickel lattice. This phenomenon is called hydrogen embrittlement and leads to a weaker structure. [8] , [14]. As a result of these conditions, the electroplating in that region is significantly slower relative to the rest of the plating area and weaker structurally.

To alleviate this problem, the plating jig holding the substrate at an angle as shown in Figure 4-8 was fabricated to allow the hydrogen gas to escape. This plating jig was made such that the plating surface was at an angle of approximately thirty degrees off vertical. Figure 4-9 and Figure 4-10 shows the hydrogen gas escaping the outer polymer clamp.

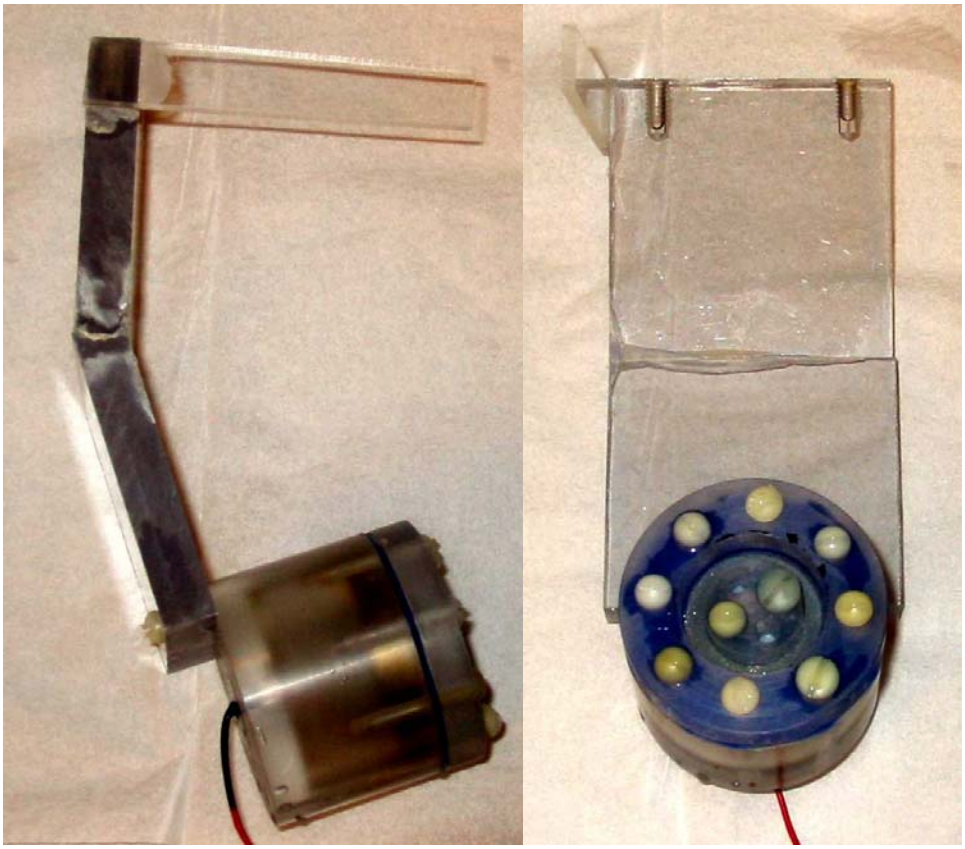

Figure 4-8: Actual Modified Plating Jig with Substrate Jig Mounted

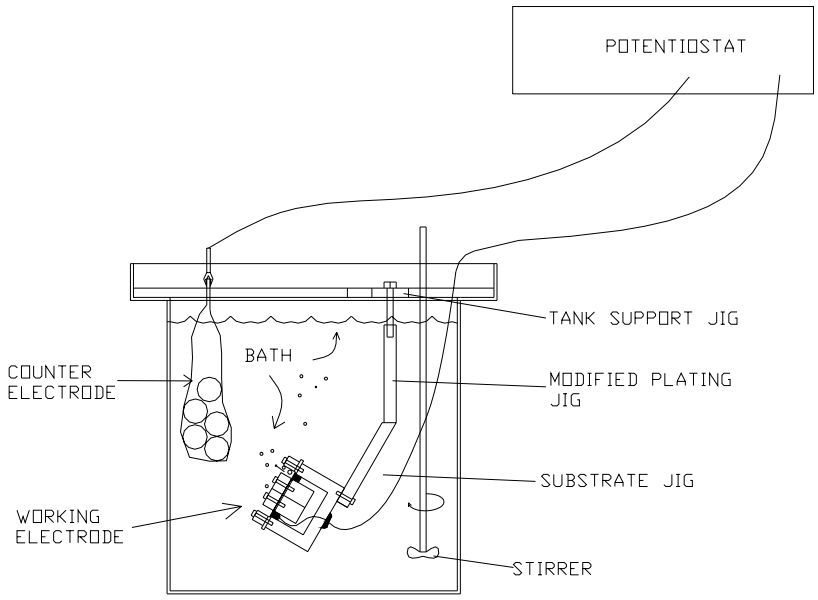

Figure 4-9: Plating Jig Modifications and Hydrogen Gas Release

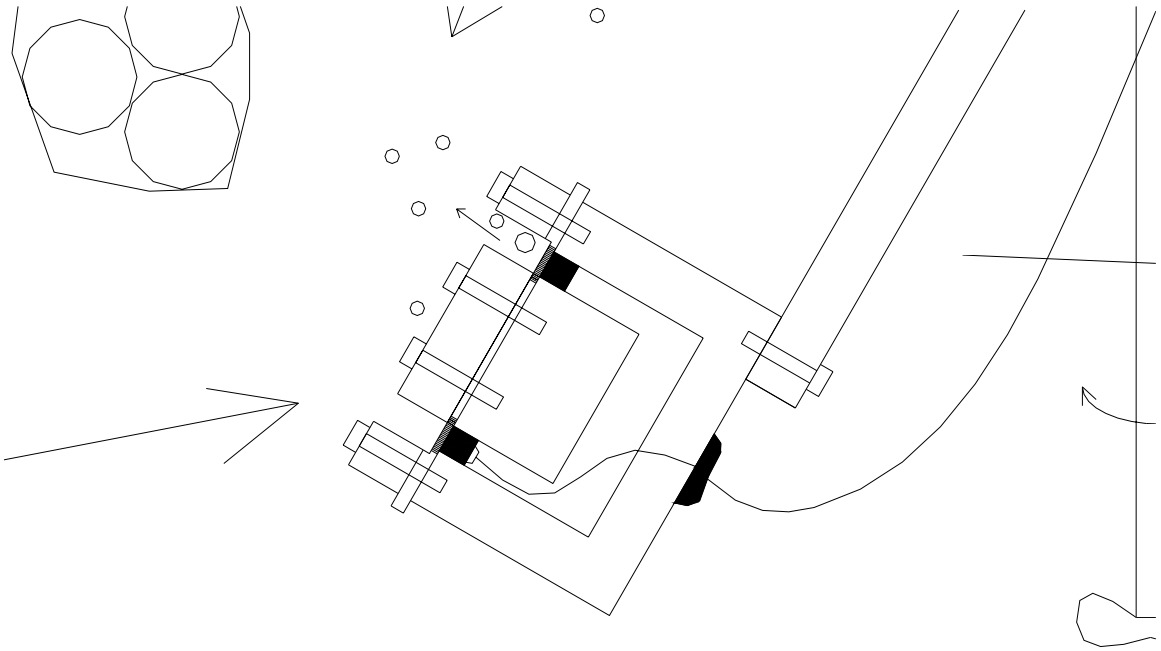

Figure 4-10: Magnified View of Plating Jig Modifications and Hydrogen Gas Release

4.5. Seal Machining Steps

In order to machine the mechanical seal, the seal needed to be separated from the substrate jig. To accomplish this, the following steps are taken. Once the electroplating is completed, the plating jig and substrate jig are removed from the electroplating solution and rinsed with DI water. The jigs are then unbolted from each other. The bolts are removed from the substrate's polymer clamps. The remaining portions of the substrate jig are then heated in an oven at 80ºC for twenty minutes. A screwdriver is then used as a wedge to separate the outer polymer clamp from the outer polymer case and mechanical seal.

Next, the outer polymer case is then removed. The electrical wire protruding from the back of the polymer case is cut and the liquid electrical tape is peeled away. An air hose blows high-pressure air into the through hole designated for the electrical wire. The increased air pressure forces the mechanical seal to translate and eventually separate from the outer polymer casing. The two polymer parts that are screwed together and define the ID of the electroplated annulus cannot be separated after the plating process is completed. These two polymer parts are removed on a lathe with a boring tool that is slightly smaller than the diameter of the polymer parts. After boring, any remaining polymer was manually removed with pliers.

At this point the seal is completely free of the substrate jig and ready to have its surfaces machined to specifications. The polymer clamps that defined the dimensions (ID and OD) of the overplated nickel were sized such that the electroplated nickel's annulus was wider than the stainless steel ring annulus as shown in Figure 4-11. To accomplish this feature, the inner polymer clamp's OD was sized 1/16" less than the

stainless steel ring's ID, and the outer polymer's ID was increased 1/16" more than the stainless steel ring's OD. The purpose of these modifications was to ensure that the polymer clamps did not cover any of the substrate's plating area when the polymer clamps were bolted to the polymer cases. If the polymer clamps did cover this area, then the electroplated nickel would not match the outer diameter of the stainless steel ring as specified. Therefore, the electroplated nickel annulus was allowed to be larger than the stainless steel ring. The additional nickel was machined down until the nickel diameters matched the stainless steel ring diameters.

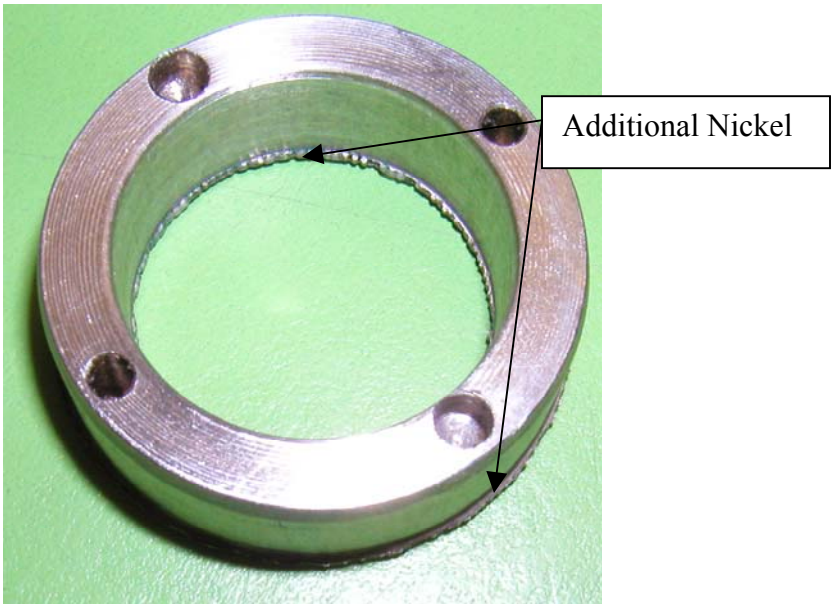

Figure 4-11: Additional Nickel on the Stainless Steel Ring's ID and OD

As stated previously, the electroplating process does not provide the required flat surface for the seal. Figure 4-12 shows a typical seal surface generated through the electroplating process. This overplated nickel was also machined to provide a flat surface and overplated nickel thickness.

Figure 4-12: Typical Seal Top Surface after Eletrodeposition

To machine the seal, a jig is built to hold the seal. The first step was to purchase a machining collet. The collet was machined on the end that typically grabs the shaft of the cutting bits that are placed into mills, lathes, etc. A cylindrical cavity was machined in this collet with a diameter slightly larger than the substrate's outer diameter and a depth approximately ¼" as shown in Figure 4-13. Figure 4-14 shows the seal resting in the cavity. Please note that the cavity depth is shallow enough to allow the outer diameter of the seal to be machined.

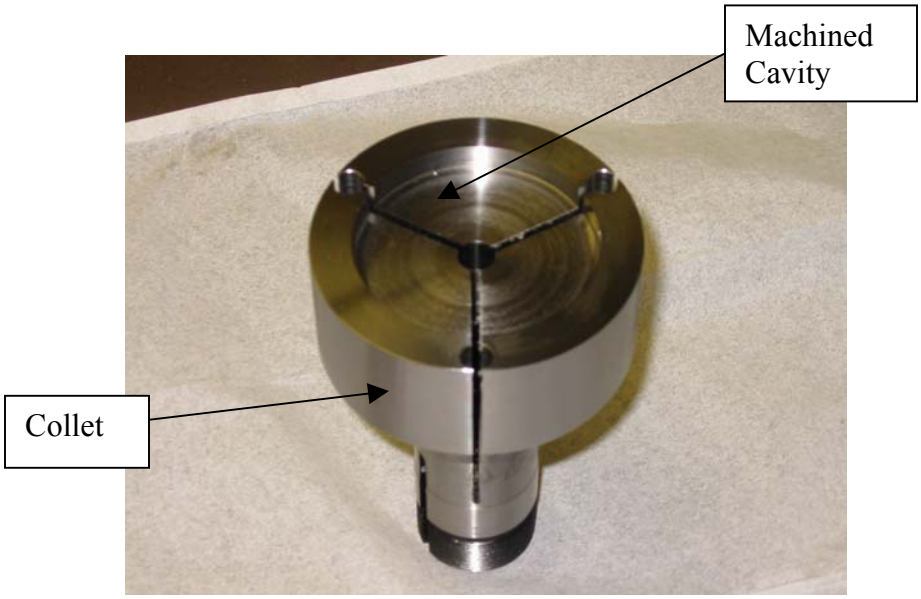

Figure 4-13: Collet with Machined Cavity

Figure 4-14: Seal Partially Machined and Held by Machined Collet

When the collet is mounted onto the lathe, the lathe's jaws clamp onto the collet. As a result, the collet clamps onto the seal. This provides a stable jig for the seal to be held while being machined on the lathe. The excess overplated nickel is then faced off to provide the desired overplated height and flat surface. The interior diameter of the seal is also bored out until the stainless steel ring's and electroplated nickel's interior diameters were equal. desired diameter is met. This process is repeated for the outer diameter. Once the machining is performed, the remaining wax template is burned out in a furnace at 500ºC for four hours.

CHAPTER 5. RESULTS AND CONCLUSIONS

5.1. Results

A process was developed to produce templates for the electroforming a LIGA heat exchanger onto a mechanical seal. Furthermore this process only required a single use of a synchrotron radiation source and produced a seal with greater bond strength. This process also proved to be easily adaptable and was used to produce a mechanical seal with larger diameters, a different micro geometrical pattern, and microposts1000 μm tall.

The result of the process developed and presented in this thesis is a mechanical seal with a post height of 650 μm at the present time. This height was limited by the height of mold insert mesh used to produce the silicone mold. The mold insert mesh's height is 950 μm. The difference in the height achieved occurred due to the machining of the wax template. To achieve micro posts of 1000 μm for this mechanical seal, a mold insert with a taller mesh needs to be used and the wax template needs to be machined more accurately.

The bond strength of the heat exchanger and substrate was improved from previous efforts. The improved bond strength due having a sandblasted surface, an underplated nickel layer, and low current density during electroplating. Eliminating the substrate's passive oxide layer and thorough activating before electroplating were also important factors in improving the bond strength. Fabricating a plating jig to limit hydrogen embrittlement was an important dynamic as well. Figure 5-1 through Figure 5-3 show different views of the fabricated seal for the process developed and presented in this thesis.

Figure 5-1: Top View of the Mechanical Seal's Nickel Surface

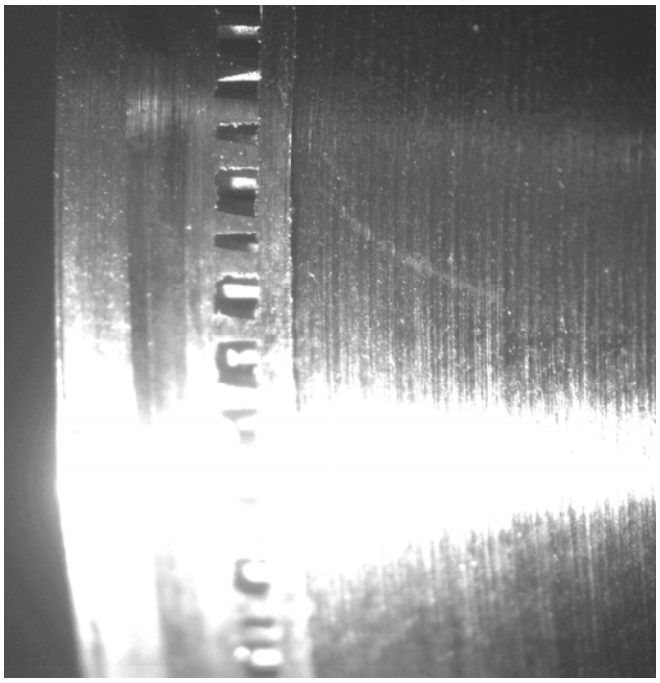

Figure 5-2: Side View of Seal's Outer Diameter

Figure 5-3: View of the Seal's Inner Diameter

Machining on the inner and outer diameters tended to smear the micro posts geometry. Removing the wax and inserting a harder filler, such as the acrylic cement, before machining the diameters would likely alleviate this problem.

5.2. Conclusions

This investigation of creating a fabrication process for the rapid production of a LIGA heat exchanger bonded to a mechanical seal with the single use of the synchrotron radiation source led to several interesting conclusions.

- A fabrication process consisting of silicone casting, machine wax casting, and electroforming can result in producing a LIGA heat exchanger bonded to a mechanical seal with the single use of a synchrotron radiation source.
- The fabrication process, once honed, produced a satisfactory template for the electroforming of a micro heat exchanger bonded to a mechanical seal.

The seal created possessed a well bonded and defined micro heat exchanger. The seal met the specifications for further thermodynamic testing to verify the effect of the micro heat exchanger lowering the seal face temperatures.

- The fabrication process has a potential for mass production that is limited by the speed of the electroplating process. Multiple silicone molds can be produced from one LIGA produced insert without any degradation to the original, and therefore, only requiring a single use of the synchrotron radiation source. Multiple templates can be produced from one silicone mold as well. With the combination of multiple silicone molds, the template production could be dramatically increased to meet any practical demands. Another major advantage is that traditional machining is all that is required during the process. This allows template production to be performed at any facility with a traditional machine shop once the LIGA mold is created. Using traditional machining also prevents the need of purchasing additional equipment and training or sub-contacting work to another machining facility.
- The adhesion between the micro heat exchanger and seal was strengthened through a series of surface treatments and electroplating conditions. The surface treatments included the use of sandblasting, activation, and Wood's strike procedure. On the electroplating side, the electroplating jig set the electroforming surface at an inclined orientation. At this

orientation, the hydrogen embrittlement was limited.. Low current density was key to providing higher adhesion strength as well.

- While the fabrication process was deemed quite successful, there are recommendations. The first is to provide a method to align the substrate jig bolt holes in the same orientation each time the jig is assemble. The second is to produce a LIGA field of structures that would produce a set bolthole orientation in the template to match the set bolthole orientation of the substrate jig. This step would bypass the entire bolt-drilling step and lead to significant time savings. The third is investigating wax additives to produce less fragile templates. Finally, removing the wax template and inserting a harder filler, such as the acrylic cement, before machining the outer and inner diameters of the seal could lead to improved micro post definition along these diameters.

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VITA

Jason Patrick Tuma was born in Monroe, Louisiana on June 5, 1977. He is the son of Frieda and Charles A. Tuma, Junior, and has an older brother, Derrick. Jason was raised in Monroe and lived there until his family moved to Pineville, Louisiana after his freshman year in high school. He graduated from Pineville High in May of 1995 and went on to attend the University of Louisiana at Monroe. In the fall of 1997, Jason began attending Louisiana State University majoring in mechanical engineering. He graduated with a Bachelor of Science in Mechanical Engineering degree with concentrations in environmental engineering and energy engineering in December 2000. He received his Master of Science in Mechanical Engineering degree from Louisiana State University in August of 2003.